SGS ACCUTEST

Quality Systems Manual

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(b) (6) [redacted]

(b) (6) [redacted] Laboratory Director, Technical Director

(b) (6) [redacted]

(b) (6) [redacted] Corporate Technical Director

(b) (6) [redacted]

(b) (6) [redacted], Quality Assurance Officer

SGS Accutest - Orlando.
4405 Vineland Road, Suite C-15
Orlando, Florida 32811
407.425.6700

INTRODUCTION

The SGS Accutest - Orlando, Inc. (SASE) Quality Assurance Program, detailed in this plan, has been designed to meet the quality program requirements of the National Environmental Laboratories Accreditation Conference (TNI), DoD QSM Ver 5.0, 2013 and ISO 17025. The plan establishes the framework for documenting the requirements of the quality processes regularly practiced by the Laboratory. The Quality Assurance Officer is responsible for changes to the Quality Assurance Program, which are appended to the Laboratory Quality Systems Manual (LQSM) as they occur. The plan is reviewed annually for compliance purposes by the Laboratory Director and Technical Director and edited if necessary. Changes that are incorporated into the plan are summarized in the plan introduction. Changes to the plan are communicated to the general staff in a meeting conducted by the Quality Assurance Officer following the plan's approval.

The SASE plan is supported by standard operating procedures (SOPs), which provide specific operational instructions on the execution of each quality element and assure that compliance with the requirements of the plan are achieved. SASE employees are responsible for knowing the requirements of the SOPs and applying them in the daily execution of their duties. These documents are updated as changes occur and the staff is trained to apply the changes.

At SGS Accutest - Orlando, we believe that satisfying client requirements and providing a product that meets or exceeds the standards of the industry is the key to a good business relationship. However, client satisfaction cannot be guaranteed unless there is a system that assures the product consistently meets its design requirements and is adequately documented to assure that all procedural steps are executed and are traceable.

This plan has been designed to assure that this goal is consistently achieved and the SGS Accutest –Orlando product withstands the rigors of scrutiny that are routinely applied to analytical data and the processes that support its generation.

SGS Accutest - Orlando is a permanent location facility and is part of SGS Accutest Inc.

Summary of Changes
SGS Accutest - Orlando Quality System Manual –March 2016

Section	Description	Page #
Title Page	new revision number	Title
Title Page	Added “Corporate” to Mr. Farmer’s TD title	Title
OrgChart	Listed Charles Hartke as Corporate QA Director	8
Entire Document	Replaced Accutest Southeast with SGS Accutest – Orlando. Replaced Accutest logo with SGS logo	
2.1; 2.2	Added three laboratories and editorial in second paragraph.	6, 7
3.3	Data integrity agreement replaced with current wording and moved to Appendix VI	14,15
6.8	Discussed SGS Accutest university database	27
4.3	SGS Accutest University database name corrected	20
10.5	Linear range requirement edited	43
19.3	Initial Orientation clarification	69
App II	Added EPA 537MOD in NPW and SCM; added 6010D.	Entire section
App III	Updated instrumentation list	87
App IV	Updated USDA permit number	92
App V	Updated SOP list	94

Table of Contents

Appendices

1.0 QUALITY POLICY

1.1 *SGS Accutest - Orlando Mission*:

SGS Accutest - Orlando provides analytical services to commercial and government clients in support of environmental monitoring and remedial activities as requested. The Laboratory's mission is dedicated to providing reliable data that satisfies the client's requirements as explained in the following: "Provide easy access, high quality, analytical support to commercial and government clients which meet or exceeds data quality objectives and provides them with the data needed to satisfy regulatory requirements and/or make confident decisions on the effectiveness of remedial activities."

These services are provided impartially and are not influenced by undue commercial or financial pressures, which might impact the staff's technical judgment. Coincidentally, SGS Accutest - Orlando does not engage in activities that endanger the trust in our independent judgment and integrity in relation to the testing activities performed.

1.2 *Policy Statement*:

The management and staff of SGS Accutest - Orlando share the responsibility for product quality and continually strive for its systematic improvement. Accordingly, SGS Accutest's quality assurance program is designed to assure that all processes and procedures, which are components of environmental data production, meet established industry requirements, are adequately documented from a procedural and data traceability perspective, and are consistently executed by the staff. It also assures that analytical data of known quality, meeting the quality objectives of the analytical method in use and the data user's requirements, is consistently produced in the laboratory. This assurance enables the data user to make rational, confident, cost-effective decisions on the assessment and resolution of environmental issues.

The laboratory Quality System also provides the management staff with data quality and operational feedback information. This enables them to determine if the laboratory is achieving the established quality and operational standards, which are dictated by the client or established by regulation, such as TNI, ISO 17025 or DoD QSM. The information provided to management, through the QA program, is used to assess operational performance from a quality perspective and to perform corrective action as necessary.

All employees of SGS Accutest - Orlando participating in environmental testing receive quality system training and are responsible for knowing and complying with the system requirements. The entire staff shares SGS Accutest's commitment to good professional practice.

(b) (6) [redacted]

Laboratory Director

2.0 ORGANIZATION

2.1 ***Organizational Entity***. SGS Accutest - Orlando, Inc. is a testing laboratory founded in 1956 and registered as a New Jersey Corporation. In 2016 the laboratory has changed ownership to SGS Accutest Inc, while operations, staff and physical locations were not affected by the change. The laboratory network headquarters are located in Dayton, New Jersey where it has conducted business since 1987. Satellite laboratories are maintained in Marlborough, Massachusetts; Syracuse, New York; Wilmington, North Carolina; Anchorage, Alaska; Orlando, Florida; San Jose, California; Denver, Colorado; Lafayette, Louisiana; and Houston, Texas.

Legal designations of the individual facilities follow SGS Accutest – Location convention, i.e. SGS Accutest – Orlando. Legal designation of the laboratory must be used on all certification and licensure documentation. Due to decades of Accutest brand prominence in their respective regions it is acceptable to display former regional designation on documents other than certificates and licenses. Example – data report from Orlando facility may be branded SGS Accutest Southeast. These documents may also use SASE abbreviation.

2.2 ***Management Responsibilities***

Requirement: Each laboratory facility will have an established chain of command. The duties and responsibilities of the management staff are linked to the President/CEO of SGS Accutest Inc. who establishes the agenda for all company activities.

President/CEO. Primarily responsible for all operations and business activities. Delegates authority to laboratory directors, laboratory managers, and quality assurance director to conduct day-to-day operations and execute quality assurance duties. Each of the individual operational entities (Massachusetts, New York, North Carolina, Alaska, Florida, California, Colorado, Louisiana, and Texas) reports to the President/CEO.

Corporate Quality Assurance Director. Responsible for design, oversight, and facilitation of all quality assurance activities established by the Quality Program. Directly reports to the President/CEO.

Chief Information Officer: Maintains and develops LIMS and various EDD formats to suit clients' requests. Maintains cyber security and confidentiality. Delegates daily LIMS operation to local labs.

Laboratory Director. A Laboratory Director is assigned to each of the following operational entities: New York, Alaska, North Carolina, New Jersey, Massachusetts Florida, Louisiana, Texas, California, and Colorado. The Laboratory Director executes day-to-day responsibility for laboratory operations including technical aspects of production activities and associated logistical procedures. The Laboratory Director reports directly to the President/CEO.

Quality Assurance Officer *(on location).* Responsible for oversight, implementation and facilitation of all quality assurance activities established by the Quality Program. Directly reports to the Laboratory Director. Also exchanges information with and submits laboratory performance data (PE scores, audit reports, accreditation changes, etc.) to Corporate QA Director. Takes program directions from Corporate QA Director.

Technical Director. Responsible for oversight and implementation of all technical aspects of production activities in the environmental testing laboratory, including method development and compliance. Directly reports to the Laboratory Director.

In the event that the technical director, quality assurance officer, or laboratory director is absent for a period of time that exceeds 15 consecutive calendar days, the designated appointees shall temporarily perform the technical director, quality assurance officer's, or laboratory director's job function. If this absence exceeds 65 consecutive calendar days, the Accreditation Body(ies), including DoD ELAP, will be notified in writing. Current list of appointed deputies located in restricted access controlled document directory.

Project Manager/Customer Service Manager: primary contact for clients requesting laboratory services. Evaluates and processes client specifications for routine and non-routine analytical services. Identifies, evaluates, and documents the requested specifications to determine if adequate resources are available to perform the analysis. Communicates the specifications to the laboratory staff for execution and verifies that specifications have been executed.

Purchasing Manager: Evaluates and vendors of services and supplies following established policies. Procures services and supplies. Maintains purchasing documentation and communicates to technical personnel prior to release into lab.

Department Supervisors. Executes day-to-day responsibility for specific laboratory areas including technical aspects of production activities and associated logistical procedures. Directly reports to the Technical Director and Laboratory Manager.

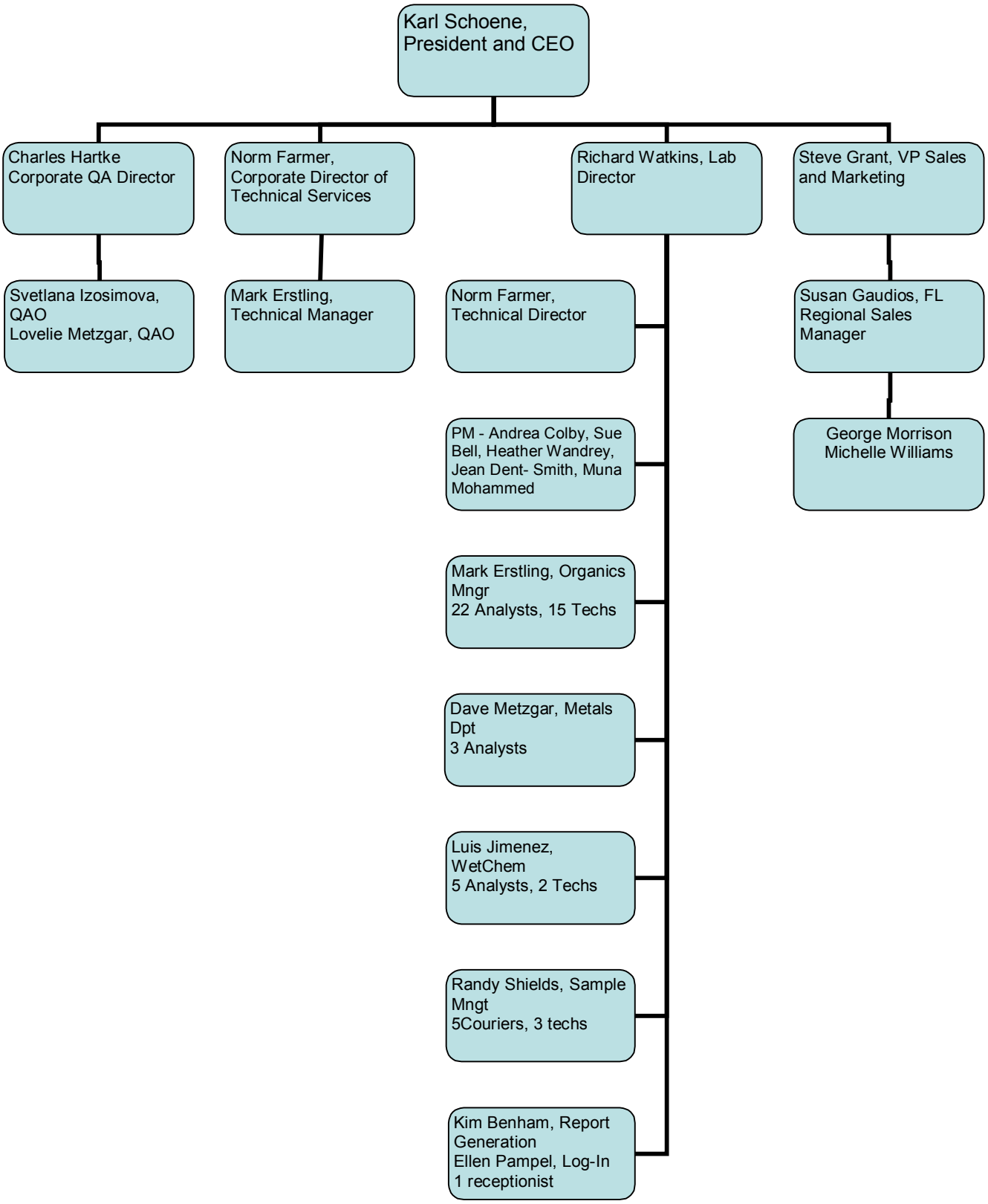
Section Supervisors. Executes day-to-day responsibility for specific laboratory units including technical aspects of production activities and associated logistical procedures. Directly report to the Department Supervisor.

2.3 ***Chain of Command***

The responsibility for managing all aspects of the Company's operation is delegated to specific individuals, who have been assigned the authority to act in the absence of the senior staff. These individuals are identified in the following Chain of Command:

Rick Watkins, Laboratory Director (Operations)
Norm Farmer, Corporate Technical Director (Operations and IT)
Heather Wandrey, Project Manager (Client Services)

SGS Accutest - Orlando Organizational Chart



3.0 QUALITY RESPONSIBILITIES OF THE MANAGEMENT TEAM

3.1 ***Requirement*:** Each member of the management team has a defined responsibility for the Quality Program. Program implementation and operation is designated as an operational management responsibility. Program design and implementation is designated as a Quality Assurance Responsibility.

President/CEO*:* Primary responsibility for all quality activities. Delegates program responsibility to the Quality Assurance Director. Serves as the primary alternate in the absence of the Quality Assurance Director. Has the ultimate responsibility for implementation of the Quality Program.

Laboratory Director*.* Responsible for implementing and operating the Quality Program in all laboratory areas. Responsible for the design and implementation of corrective action for defective processes. Has the authority to delegate Quality Program implementation responsibilities.

Corporate Quality Assurance Director*.* Responsible for design, implementation support, training, and monitoring of the quality system. Identifies product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Empowered with the authority to halt production if warranted by quality problems. Monitors implemented corrective actions for compliance.

Quality Assurance Officer *(on location).* Responsible for design support, implementation support, and monitoring support of the quality system. Training personnel in various aspects of quality system. Conducts audits and product reviews to identify product, process, or operational defects using statistical monitoring tools and processes audits for elimination via corrective action. Empowered with the authority to halt production if warranted by quality problems. Monitors implemented corrective actions for compliance.

Technical Director. Responsible for oversight and implementation of technical aspects of Quality System as they are integrated into method applications and employed to assess analytical controls on daily basis. The Technical Director reviews and acknowledges the technical feasibility of proposed quality system involving technical applications. Empowered with the authority to halt production if warranted by quality problems.

Laboratory Manager. Responsible for oversight and implementation of various aspects of Quality System as they are integrated into method applications and employed to assess analytical controls on daily basis. The Laboratory Manager reviews and acknowledges the technical and logistical feasibility of proposed quality system involving technical applications. Empowered with the authority to halt production if warranted by quality problems.

Department Supervisors. Responsible for applying the requirements of the Quality Program in their section and assuring subordinate supervisors and staff apply all program requirements. Initiates, designs, documents, and implements corrective action for quality deficiencies.

Section Supervisors. Responsible for applying the requirements of the Quality Program to their operation and assuring the staff applies all program requirements. Initiates, designs, documents, and implements corrective action for quality deficiencies.

Bench Analysts. Responsible for applying the requirements of the Quality Program to the analyses they perform, evaluating QC data and initiating corrective action for quality control deficiencies within their control. Implements global corrective action as directed by superiors.

3.2 ***Program Authority***:

Authority for program implementation on corporate level originates with the President/CEO who bears ultimate responsibility for program design, implementation, and enforcement of requirements. This authority and responsibility is delegated to the Director of Quality Assurance who performs quality functions independently without the encumbrances or biases created by operational or production responsibilities to ensure an honest, independent assessment of quality issues.

Laboratory Director and Quality Assurance Officer mirror this authority on location.

3.3 ***Data Integrity Policy***:

The SGS Accutest Data Integrity Policy reflects a comprehensive, systematic approach for assuring that data produced by the laboratory accurately reflects the outcome of the tests performed on field samples and has been produced in a bias free environment by ethical professionals. The policy includes a commitment to technical ethics, staff training in ethics and data integrity, an individual attestation to data integrity and procedures for evaluating data integrity. Senior management assumes the responsibility for assuring compliance with all technical ethics elements and operation of all data integrity procedures. The staff is responsible for compliance with the ethical code of conduct and for practicing data integrity procedures.

The SGS Accutest Data Integrity Policy is as follows:

"SGS Accutest - Orlando is committed to producing data that meets the data integrity requirements of the environmental regulatory community. This commitment is demonstrated through the application of a comprehensive data integrity program that includes ethics and data integrity training, data integrity evaluation procedures, staff participation and management oversight. Adherence to the specifications of the program assures that data provided to our clients is of the highest possible integrity and can be used for decision making processes with high confidence."

Data Integrity Responsibilities

Management. Senior management retains oversight responsibility for the data integrity program and retains ultimate responsibility for execution of the data integrity program elements. Senior management is responsible for providing the resources required to conduct ethics training and operate data integrity evaluation procedures. They also include responsibility for creating an environment of trust among the staff and being the lead advocate for promoting the data integrity policy and the importance of technical ethics.

Staff. The staff is responsible for adhering to the company ethics policy as they perform their duties and responsibilities associated with sample analysis and reporting. By executing this responsibility, data produced by SGS Accutest retains its high integrity characteristics and withstands the rigors of all data integrity checks.

The staff is also responsible for adhering to all laboratory requirements pertaining to manual data edits, data transcription and data traceability. These include the application of approved manual peak integration and documentation procedures. It also includes establishing traceability for all manual results calculations and data edits.

Ethics Statement. The SGS Accutest ethics statement reflects the standards that are expected for businesses that provide environmental services to regulated entities and regulatory agencies on a commercial basis. The Ethics Policy is comprised of key elements that are essential to organizations that perform chemical analysis for a fee. As such, it focuses on elements related to personal, technical and business activities.

SGS Accutest - Orlando provides analytical chemistry services on environmental matters to the regulated community. The data the company produces provides the foundation for determining the risk presented by a chemical pollutant to human health and the environment. The environmental industry is dependent upon the accurate portrayal of environmental chemistry data. This process is reliant upon a high level of scientific and personal ethics.

It is essential to the Company that each employee understands the ethical and quality standards required to work in this industry. Accordingly, SGS Accutest has adopted a code of ethics, which each employee is expected to adhere to as follows:

- Perform chemical and microbiological analysis using accepted scientific practices and principles.
- Perform tasks in an honest, principled and incorruptible manner inspiring peers & subordinates.
- Maintain professional integrity as an individual.
- Provide services in a confidential, honest, and forthright manner.
- Produce results that are accurate and defensible.

- Report data without any considerations of self-interest.
- Comply with all pertinent laws and regulations associated with assigned tasks and responsibilities.

Data Integrity Procedures.

Four key elements comprise the SGS Accutest data integrity system:

1) data integrity training,

2) signed data integrity documentation for all laboratory employees,

3) in-depth, periodic monitoring of data integrity, and

4) data integrity procedure documentation.

Procedures have been implemented for conducting data integrity training and for documenting that employees conform to the SGS Accutest Data Integrity and Ethics policy.

The data integrity program consists of routine data integrity evaluation and documentation procedures to periodically monitor and document data integrity. These procedures are documented in SOPs. SOPs are approved and reviewed annually following the procedures employed for all SGS Accutest SOPs. Documentation associated with data integrity evaluations is maintained on file and is available for review.

Data Integrity Training. SGS Accutest employees receive technical ethics training during new employee orientation. Employees are also required to attend annual ethics refreshment training and sign an ethical conduct agreement annually, which verifies their understanding of SGS Accutest's technical ethics policy and their ethical responsibilities. The agreement is refreshed annually and appended to each individual's training file.

The training focuses on the reasons for technical ethic training, explains the impact of data fraud on human health and the environment, and illustrates the consequences of criminal fraud on businesses and individual careers. Multiple examples of prohibited practices are reviewed and discussed. SGS Accutest's ethics policy and code of ethics are reviewed and explained for each new employee. Employees receive SGS Accutest's technical ethics brochure for further review.

Training on department-specific data integrity procedures are conducted by individual departments for groups involved in data operations. These include procedures for manual chromatographic peak integration, standards traceability, etc.

Data Integrity Training Documentation. Records of all data integrity training are maintained in individual training folders. Attendance at all training sessions is documented and appended to the training file.

SGS Accutest Data Integrity and Ethical Conduct Agreement. All employees are required to sign a Data Integrity and Ethical Conduct Agreement annually – See Appendix VI This document is archived in individual training files, which are retained for duration of employment.

Data Integrity Monitoring. Several documented procedures are employed for performing data integrity monitoring. These include regular data review procedures by supervisory and management staff (Section 12.7), supervisory review and approval of manual integrations and periodic reviews of data audit trails from the LIMS and all computer controlled analysis.

Data Review. All data produced by the laboratory undergoes several levels of review, which includes two levels of management review. Detected data anomalies that appear to be related to data integrity issues are isolated for further investigation. The investigation is conducted following the procedures described in this section.

Manual Peak Integration Review and Approval. Routine data review procedures for all chromatographic processes includes a review of all manual chromatographic peak integrations. This review is performed by the management staff and consists of a review of the machine integration compared to the manual integration. Manual integrations, which have been performed in accordance with SGS Accutest's manual peak integration procedures are approved for further processing and release. Manual integrations which are not performed to SGS Accutest's specifications are set aside for corrective action, which may include analyst retraining or further investigation as necessary.

Data Audit Trail Review. Data integrity audits are comprehensive data package audits that include a review of raw data, process logbooks, processed data reports and data audit trails from individual instruments and LIMS. Data audit trails, which record all electronic data activities, are available for the majority of computerized methodology and the laboratory information management system (LIMS). These audit trails are periodically reviewed to determine if interventions performed by technical staff constitute an appropriate action. The review is performed on a recently completed job and includes interviews with the staff that performed the analysis. Findings indicative of inappropriate interventions or data integrity issues are investigated to determine the cause and the extent of the anomaly.

Confidential Reporting Of Data Integrity Issues. Data integrity concerns may be raised by any individual to their supervisor. Employees with data integrity concerns should always discuss those concerns with their immediate supervisors as a first step unless the employee is concerned with the confidentiality of disclosing data integrity issues or is uncomfortable discussing the issue with their immediate supervisors. The supervisor makes an initial assessment of the situation to determine if the concern is

related to a data integrity violation. Those issues that appear to be violations are documented by the supervisor and referred to the QA Officer (local) for investigation.

Documented procedures for the confidential reporting of data integrity issues in the laboratory are part of the data integrity policy. These procedures assure that laboratory staff can privately discuss ethical issues or report items of ethical concern without fears of repercussions with senior staff.

Employees with data integrity concerns that they consider to be confidential are directed to the Corporate Human Resources Manager in Dayton, New Jersey. The HR Manager acts as a conduit to arrange a private discussion between the employee and the Corporate QA Director or a local QA Officer.

During the employee - QA discussion, the QA representative evaluates the situation presented by the employee to determine if the issue is a data integrity concern or a legitimate practice. If the practice is legitimate, the QA representative clarifies the process for the employee to assure understanding. If the situation appears to be a data integrity concern, the QA representative initiates a Data Integrity Investigation following the procedures specified in SOPs QA038-QA041.

Data Integrity Investigations. Follow-up investigations are conducted for all reported instances of ethical concern related to data integrity. Investigations are performed in a confidential manner by senior management according to a documented procedure. The outcome of the investigation is documented and reported to the company president who has the ultimate responsibility for determining the final course of action in the matter. Investigation documentation includes corrective action records, client notification information and disciplinary action outcomes, which is archived for a period of five years.

The investigations are conducted by the senior staff and supervisory personnel from the affected area. The investigation team includes the Laboratory Director and the Quality Assurance Officer. Investigations are conducted in a confidential manner until it is completed and resolved.

The investigation includes a review of the primary information in question by the investigations team. The team performs a review of associated data and similar historical data to determine if patterns exist. Interviews are conducted with key staff to determine the reasons for the observed practices.

Following data compilation, the investigations team reviews all information to formulate a consensus conclusion. The investigation results are documented along with the recommended course of action.

Corrective Action, Client Notification & Discipline. Investigations that reveal systematic data integrity issues will go through corrective action for resolution and disposition (Section 13). If the investigation indicates that an impact to data has occurred and the defective data has been released to clients, client notification procedures will be initiated following the steps in Section 17.6.

In all cases of data integrity violations, some level of disciplinary action will be conducted on the responsible individual. The level of discipline will be consistent with the violation and may range from retraining and/or verbal reprimand to termination.

4.0 JOB DESCRIPTIONS OF KEY STAFF

4.1 ***Requirement*:** Descriptions of key positions within the organization must be defined to ensure that clients and staff understand duties and the responsibilities of the management staff and the reporting relationships between positions.

President/Chief Executive Officer. Responsible for all laboratory operations and business activities. Establishes the company mission and objectives in response to business needs. Direct supervision of the Vice President of Operations, each laboratory director, client services, management information systems, and quality assurance.

Laboratory Director. Reports to the company president. Establishes regional laboratory operations strategy and business development. Authorized to enter into contractual agreements on Company's behalf. Directs the day-to-day operations of entire laboratory, direct supervision of organic chemistry, inorganic chemistry, field services, and sample management. Oversees daily work schedule as developed by respective departments. Supervises method implementation. Responsible for following Quality Program requirements. Maintains laboratory instrumentation in an operable condition.

Director, Quality Assurance. Reports to the company president. Establishes the company quality agenda, develops quality procedures, provides assistance to operations on quality procedure implementation, coordinates all quality control activities monitors the quality system and provides quality system feedback to management to be used for process improvement.

Vice President, Information Technologies Reports to the company president. Develops the MIS software and hardware agenda. Provides system strategies to compliment company objectives. Maintains all software and hardware used for data handling.

Client Services, Sales, Account Manager(s). Reports to the company president. Establishes and maintains communications between clients and the laboratory pertaining to client requirements which are related to sample analysis and data deliverables. Initiates client orders and supervises sample login operations.

Quality Assurance Officer *(on location)*. Reports to the Corporate QA Director. Develops quality procedures, provides assistance to operations on quality procedure implementation, coordinates all quality control activities, monitors the quality system, and provides quality system feedback to management to be used for process improvement. In the event of prolonged absence QAO also designated a Deputy Technical Director, unless otherwise specified by internal memo from Laboratory Director.

Manager Client Services *(on location)*. Reports to the Laboratory Director. Establishes and maintains communications between clients and the laboratory

pertaining to client requirements which are related to sample analysis and data deliverables. Initiates client orders and supervises sample login operations.

Technical Director (*on Location***)**. Reports to the Laboratory Director. Establishes laboratory operations strategy. Direct supervision of organic chemistry and inorganic chemistry. Directs the operations, preparation and instrumental analysis. Responsible for following Quality Program requirements. Assumes operational responsibilities of Lab Director in his absence.

Supervisors, Shipping and Receiving Departments. Reports to the Laboratory Director. Develops, maintains and executes all procedures required for transport and receipt of samples, verification of preservation, and chain of custody documentation. Responsible for maintaining and documenting secure storage, delivery of samples to laboratory units on request, and disposal following completion of all analytical procedures.

Supervisor, Wet Chemistry. Reports to the Laboratory Director. Directs the operations of the wet chemistry group. Establishes and executes daily work schedule. Supervises method implementation, application, and data production. Supervises the analysis of samples for wet chemistry parameters using valid, documented methodology. Maintains instrumentation in an operable condition. Reviews data for compliance to quality and methodological requirements. Responsible for following Quality Program requirements.

Supervisor, Metals. Reports to the Laboratory Director. Directs the operations of the metals group. Establishes and executes daily work schedule. Supervises method implementation, application, and data production. Supervises the analysis of samples for metallic elements using valid, documented methodology. Documents all procedures and data production activities. Maintains instrumentation in an operable condition. Reviews data for compliance to quality and methodological requirements. Responsible for following Quality Program requirements

Supervisor, Organic Preparation. Reports to the Laboratory Director. Directs the operations of the sample preparation group. Establishes and executes daily work schedule. Supervises method implementation, and application. Supervises the preparation of samples for organic compounds using valid, documented methodology. Documents all procedures and data production activities. Maintains laboratory equipment in an operable condition. Reviews records for compliance to quality and methodological requirements. Responsible for following Quality Program requirements.

Volatile and Semivolatie Supervisors, Organics. Reports to the Laboratory Director. Directs the operations of the respective organics group. Establishes and executes daily work schedule. Supervises method implementation, application, and data production. Supervises the analysis of samples for organic compounds using valid, documented methodology. Documents all procedures and data production activities. Maintains instrumentation in an operable condition. Reviews data for compliance to

quality and methodological requirements. Responsible for following Quality Program requirements

Report Generation Supervisor. Reports to Laboratory Director. Oversees report generation and fulfillment of client specifications as applied to data deliverables. Responsible for data delivery in timely manner.

Detailed Job descriptions of lab personnel are found in training folders

4.2 ***Employee Screening, Orientation, and Training***.

All potential laboratory employees are screened and interviewed by human resources and technical staff prior to their hire. The pre-screen process includes a review of their qualifications including education, training and work experience to verify that they have adequate skills to perform the tasks of the job. Minimum qualifications for non-technical personnel require High School diploma (couriers also must posses clean driving record), technical personnel must also demonstrate basic laboratory experience, such as balance and syringe use, aseptic practices, etc. College-level science coursework is favored.

Newly hired employees receive orientation training beginning the first day of employment by the Company. Orientation training consists of initial health and safety training and a detailed review of the personal protection policies, technical ethics and data integrity procedures training (for detailed description refer to Sec. 3.3) and quality assurance program training (including Company's goals, objectives, mission, and vision).

All technical staff receives training to develop and demonstrate proficiency for the methods they perform. New analysts work under supervision until the supervisory staff is satisfied that a thorough understanding of the method is apparent. Organics/Inorganics analysts are required to demonstrate method proficiency through a precision and accuracy study (Demonstration of Capability). Data from the study is reviewed by appropriate technical supervisor and compared to method acceptance limits. If the data is unacceptable, additional training is required. The analyst must also demonstrate the ability to produce acceptable data through the analysis of an independently prepared proficiency sample.

Proficiency is demonstrated annually. Data from initial and continuing proficiency demonstration is archived in the individual's training folder. In the instance where analyte can not be spiked in the clean matrix, such as TSS or pH, the results of an external Performance Evaluation (PE) sample may be used to document analyst's proficiency.

Minimum training required for administrative staff consists of laboratory safety and ethical conduct.

4.3 ***Training Documentation***. The QA Officer prepares a training file for every new employee. All information related to qualifications, experience, external training

courses, and education are placed into the file. Verification documentation for orientation, health & safety, quality assurance, and ethics training is also included in the file.

Additional training documentation is added to the file as it occurs. This includes data for initial and continuing demonstrations of proficiency, performance evaluation study data and notes and attendance lists from group training sessions.

The Quality Assurance Department also maintains the employee training database – SGS Accutest University. This database is a comprehensive inventory of training documentation for each individual employee. The database enables supervisors to obtain current status information on training data for individual employees on a job specific basis. It also enables the management staff to identify training documentation in need of completion.

Employee specific database records are created by QA Staff on the date of hire. Reports are produced which summarize the qualifications of individual employees or departments.

5.0 SIGNATORY APPROVALS

Requirement: Procedures are required for establishing the traceability of data and documents. The procedure consists of a signature hierarchy, indicating levels of authorization for signature approvals of data and information within the organization. Signature authority is granted for approval of specific actions based on positional hierarchy within the organization and knowledge of the operation that requires signature approval. A log of signatures and initials of all employees is maintained for cross-referencing purposes.

5.1 *Signature Hierarchy*.

President/Chief Executive Officer. Authorization for contracts and binding agreements with outside parties. Approval of quality assurance policy, SOPs, project specific QAPs. Contract signature authority resides with Company Officers only, which include the President/CEO, CFO and VP Administration.

Laboratory Director. Authorization for contracts and binding agreements with outside parties. Approval of final reports and quality assurance policy. Approval of project specific QAPs. Review and approval of technical and quality systems policies (LQSM). In the event of prolonged absence refer to list of approved deputies – sec 2.2.

Technical Director: Approval of final reports and quality assurance policy in the absence of the Laboratory Director. Approval of SOPs, project specific QAPs. Review and approval of technical and quality systems policies (LQSM). In the event of prolonged absence refer to list of approved deputies – sec 2.2.

Director, Quality Assurance. Approval of final reports and quality assurance policy in the absence of the President. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers.

Quality Assurance Officer (on location). Approval of final reports and quality assurance policy in the absence of the Laboratory Director. Approval of SOPs, project specific QAPs, data review and approval in lieu of technical managers. In the event of prolonged absence refer to list or appointed deputies – see sec. 2.2.

Manager, Sample Management. Initiation of laboratory sample custody and acceptance of all samples. Approval of department policies and procedures. Department specific supplies purchase. Waste manifesting and disposal.

Project Manager, Client Services. QAP and sampling and analysis plan approval. Project specific contracts, pricing, and price modification agreements. Approval and acceptance of incoming work, Client services policy.

Supervisors, Technical Departments. Methodology and department specific QAPs. Data review and approval, department specific supplies purchase. Technical approval of SOPs.

5.2 ***Signature Requirements***. All laboratory activities related to sample custody and generation or release of data must be approved using either initials or signatures. The individual, who applies his signature or initial to an activity or document, is authorized to do so within the limits assigned to them by their supervisor. All signatures and initials must be applied in a readable format that can be cross-referenced to the signatures and initials log if necessary.

5.3 ***Signature and Initials Log***. The QA Officer maintains a signature and initials log. New Employee signatures and initials are appended to the log on the first day of employment. Signature of individuals no longer employed by the company are retained**.**

6.0 DOCUMENTATION and DOCUMENT CONTROL

Requirement: Document control policies have been established which specify that any document used as an information source or for recording analytical or quality control information must be managed using defined document control procedures. Accordingly, policies and procedures required for the control, protection, and storage of any information related to the production of analytical data and the operation of the quality system to assure its integrity and traceability have been established and implemented in the laboratory. The system contains sufficient controls for managing, archiving and reconstructing all process steps, which contributed to the generation of an analytical test result. Using this system, an audit trail for reported data can be produced, establishing complete traceability for the result.

6.1 Administrative Records. The Quality Assurance Officer manages Administrative (non-analytical) records. These records consist of electronic documents that are retained in a limited access electronic directory, which are released to the technical staff upon specific request.

Form Generation & Control. The Quality Assurance Officer approves all forms used as either stand-alone documents or in logbooks to ensure their traceability. Forms are generated as computer files only and maintained in a limited access master directory. Access to the electronic forms and applications is granted to QA Officer, Laboratory Manager and Technical Director(s) (local and regional). Approved forms must display the date of current revision and initials of person who revised the form. Modifications to existing forms are approved by QA, obsolete forms moved to archive directory and retained for minimum of five years.

New forms must include SGS Accutest - Orlando identification and appropriate spaces for signatures of approvals and dates. Further design specifications are the responsibility of the originating department.

Technical staff is required to complete all forms to the maximum extent possible. If information for a specific item is unavailable, the analyst is required to cross out the information block. The staff is also required to cross out the uncompleted portions of a logbook or logbook form if the day's analysis does not fill the entire page of the form.

Logbook Control. All laboratory logbooks are controlled documents that are comprised of approved forms used to document specific processes. Logbook control is maintained by QA staff.

New logs are numbered and issued to a specific individual who is assigned responsibility for the log. Supervisor performs periodical review of the logbooks. Old logs are returned to QA for entry into the document archive system where they are retained for minimum of five (5) years. Laboratory staff may hold a maximum of two consecutively dated logbooks of the same type in the laboratory, not including the most recently issued book to simplify review of recently completed analysis.

Controlled Documents. Key laboratory documents are designated for controlled document status to assure that identities of individuals receiving copies and the number of copies that have been distributed are known. Controlled status simplifies document updates and **retrieval** of outdated documents. Control is maintained through a document numbering procedure and document control logbook designating the individual receiving the controlled document. Document control is also maintained by pre-designating the numbers of official copies of documents that are placed into circulation within the laboratory.

Quality Systems Manual (QSM). All QSMs are assigned a number prior to distribution. The QSMs are distributed as controlled documents i.e. ones that will be collected back and replaced with next version (documents distributed to the SGS Accutest Inc. staff). QSMs distributed to outside entities are considered tracked documents – since there is no possibility of collecting them back and ensuring that current revision is in use. These situations include bid submissions, client requests, etc. These copies are watermarked as "Uncontrolled Documents" The control/tracking number, date of distribution, and identity of the individual receiving the document are recorded in the document control spreadsheet. QA staff maintains tracking spreadsheet. The numbering system is continuous.

Standard Operating Procedures (SOPs). SOPs are maintained by pre-designating the numbers of official copies of documents that are placed into circulation within the laboratory. Official documents are printed and placed into the appropriate laboratory section as follows:

Sample Management: One copy for the sample receiving file
Bottle preparation area – One copy for shipping area
Organics Laboratories: One for each affected laboratory area. Copy may be electronic.
Inorganics Laboratories: One for each affected laboratory area. Copy may be electronic.

The original, signed copy of the SOP is maintained in the master SOP binder by the QA staff.

Documents are controlled using an "Official Copy" stamp in red ink. Additional copies could be issued to individuals for training purposes. Distribution is documented on SOP cover page. Superseded copies collection is conducted accordingly to cover page distribution list.

SOPs distributed to clients as part of bid submission, pre-audit evaluation, etc. are watermarked as "Proprietary Information".

Quick reference cards: These documents are compiled for lab staff convenience and are based on current SOP revision and/or recent regulatory updates. These one- or two-sided documents are footnoted with reference to SOP/regulatory standard, stamped with "Official Copy" stamp in red ink and laminated for durability. ***Use of***

these quick references does not substitute reading and acknowledging the parent SOP.

Operators' Manuals are considered controlled documents and stored in appropriate departments.

6.2 ***Technical Records***. All records related to the analysis of samples and the production of analytical results are archived in secure document storage or on electronic media and contain sufficient detail to produce an audit trail, which re-creates the analytical result. These records include information related to the original client request, bottle order, sample login and custody, storage, sample preparation, analysis, data review and data reporting.

Records that can not be maintained on electronic media are considered irretrievable records, segregated into separate secured storage and access controlled with access log maintained by QA Staff. Examples of such records are employee training files, obsolete SOPs and acknowledgement form originals, training files, logbooks, etc.

Each department involved in this process maintains controlled documents, which enable them to maintain records of critical information relevant to their department's process.

6.3 ***Quality Assurance Directory.*** All Quality Assurance documentation and quality control limit data is stored in a restricted QA directory on the network server. The directory has been designated as read only. The QA staff, technical director and the laboratory manager have write capability in this directory. Information on this directory is backed-up weekly.

This directory contains all current and archived Quality System Manuals, SOPs, control limits, MDL studies, precision and accuracy data, internal and external audit reports, official forms, Health and Safety materials, PT scores, State Certifications and metrics calibration information.

6.4 ***Analytical Records***. All data related to the analysis of field samples are retained as either paper or electronic records that can be retrieved to compile a traceable audit trail for any reported result. All information is linked to the client job and sample number, which serves as a reference for all sample related information tracking.

Critical times in the life of the sample from collection through analysis to disposal are documented. This includes date and time of collection, receipt by the laboratory, preparation times and dates, analysis times and dates and data reporting information. Analysis times are calculated in hours for methods where holding time is specified in hours (≤72 hours).

Sample preparation information is recorded in a separate controlled logbook or on controlled forms in three-ring binder. It includes sample identification numbers, types of analysis, preparation and cleanup methods, sample weights and volumes, reagent

lot numbers and volumes and any other information pertinent to the preparation procedure.

Information related to the identification of the instrument used for analysis is permanently attached to the electronic record. The record includes an electronic data file that indicates all instrument conditions employed for the analysis, including the type of analysis conducted. The analyst's identification is electronically attached to the record. The instrument tuning and calibration data is electronically linked to the sample or linked though paper logs, which were used in the documentation of the analysis. Quality control and performance criteria are permanently linked to the paper archive or electronic file.

Paper records for the identity, receipt, preparation and evaluation of all standards and reagents used in the analysis are documented in prepared records and maintained in controlled documents or files. Lot number information linking these materials to the analysis performed is recorded in the logbooks associated with the samples in which they were used.

Manual calculations or peak integrations that were performed during the data review are retained as paper or electronically generated PDF documents and included as part of the electronic archive. Signatures for data review are retained on paper or as electronic stamps on PDF versions of the paper record for the permanent electronic file.

6.5 ***Confidential Business Information (CBI).*** Operational documents including SOPs, Quality Manuals, personnel information, internal operations statistics, and laboratory audit reports are considered confidential business information. Strict controls are placed on the release of this information to outside parties.

Release of CBI to outside parties or organizations may be authorized upon execution of a confidentiality agreement between SGS Accutest and the receiving organization or individual. CBI information release is authorized for third party auditors and commercial clients in electronic mode as Adobe Acrobat .PDF format only.

6.6 ***Software Change Documentation & Control.*** Changes to software are documented as text within the code of the program undergoing change. Documentation includes a description of the change, reason for change and the date the change was placed into effect. Documentation indicating the adequacy of the change is prepared following the evaluation by the user who requested the change.

6.7 ***Report and Data Archiving***. SGS Accutest maintains electronic image file copies of original reports in archive for a minimum period of five (5) years. After five years, the files are automatically discarded unless contractual arrangements exist which dictate different requirements. Client specific data retention practices are employed for government organizations such as the Department of Defense Agencies and MA DEP that require a retention period of ten (10) years, as well as commercial clients upon contractual requirements agreement.

Complete date and time stamped client reports are generated from LIMS using the source documents archived on Document server. These source documents are maintained on document server and backed up to removable primary and clone hard drives. SGS Accutest archives the original report (organized by job number) and the organic and inorganic support data. Organic support data is archived according to instrument batch numbers and datafile. All organics data is backed up to an archive drive via Baculla backup software and/or AccuBack backup software. Data from the archive drive is then written to removable hard drives at periodic intervals. Drives are cloned for off site archival.

Wet chemistry support data is archived by analytical batch (GN…). Metals support data is archived by instrument batch (MA…). Metals digestion data is archived as digestion logbooks. Additionally all Wet chemistry and Metals data is scanned to PDF and archived electronically.

The reports generation group electronically scans completed reports and stores them by job number on the document server. The document server is backed up daily to a removable hard drive. Copies of these files remain active on the document server for easy review access. The removable hard drives remain in secure storage for the remainder of the archive period.

6.8 ***Training***. Ongoing training ensures competence of all relevant personnel. At the minimum personnel should possess knowledge of the technology used in the testing, general requirements expressed in legislature and industry standards, and understand the significance of deviations with regard to approved procedures. The company maintains a training record for all employees that documents that they have received instruction on administrative and technical tasks that are required for the job they perform. Training records for individuals employed by the company are retained for a period of five years following their termination of employment.

Training File Origination. The Quality Assurance Officer (QAO) initiates training files. Quality Assurance officer retains the responsibility for the maintenance and tracking of all training related documentation in the file. The file is started on the first day of employment. Information required for the file includes a copy of the individual's most current resume, detailing work experience and a copy of any college diplomas or transcript(s), if applicable. Information added on the first day includes documentation of health and safety training and a signed Ethics and Data Integrity agreement. Classroom content is standardized across entire SGS Accutest network and administered using SGS Accutest University database. Safety and Ethics training constitute minimal necessary training for Project Management and Administrative staff. Analyst training documentation, training requirements, analyst proficiency information and other training related support documentation is tracked using a customized database applications. Database extracts provide an itemized listing of specific training requirements by job function. Training status summaries for individual analysts portray dates of completion for job specific training requirements.

Technical Training. The supervisor of each new employee is responsible for developing a training plan for each new employee. The supervisor updates the

outline, adding signatures and dates as training elements are completed at regular frequency. Supporting documentation, such as precision and accuracy studies, which demonstrate analyst capability for a specific test, are added as completed. When analyte can not be spiked, such as pH or TSS, external PE sample is purchased and analyzed. Where no external PE sample is available, sample duplicates must be successfully analyzed. Method review records are retained where analysis of duplicates is not possible. Employees and supervisors verify documentation of understanding (DOU) for all assigned standard operating procedures in the training database. Certificates or diplomas for any off-site training are added to the file.

7.0 REFERENCE STANDARD TRACEABILITY

Requirement: Documented procedures, which establish traceability between any measured value and a national reference standard, must be in place in the laboratory. All metric measurements must be traceable to NIST reference weights or thermometers that are calibrated on a regular schedule. All chemicals used for calibration of a quantitative process must be traceable to an NIST reference that is documented by the vendor using a certificate of traceability. The laboratory maintains a documentation system that establishes the traceability links. The procedures for verifying and documenting traceability must be documented in standard operating procedures.

7.1 ***Traceability of Metric Measurements - Thermometers***. SGS Accutest – Orlando uses NIST-traceable thermometers to calibrate commercially purchased working laboratory thermometers prior to their use in the laboratory and annually thereafter for liquid in glass thermometers or quarterly for electronic temperature measuring devices. If necessary, these working thermometers are assigned correction factors that are determined during their calibration using an NIST-traceable thermometer as the standard. The correction factor is documented in a thermometer log and on a tag attached to the working thermometer. Both original observation and corrected measurement are recorded in the temperature log. The NIST-traceable reference thermometer is checked for accuracy by an outside vendor minimum every five (5) years following the specifications for NIST-traceable thermometer calibration verification detailed in the United States Environmental Protection Agency's "Manual for the Certification of Laboratories Analyzing Drinking Water", Fifth Edition, January 2005. Currently the NIST thermometer is verified by outside vendor on triennial basis due to contract-specific requirements. Calibration log and Certificate(s) of calibration are maintained on file with QAO.

7.2 ***Traceability of Metric Measurements – Calibration Weights***. SGS Accutest – Orlando uses calibrated weights, which are traceable to NIST standard weights to calibrate all balances used in the laboratory. Balances must be calibrated to specific tolerances within the intended use range of the balance. Calibration checks are required on each day of use. If the tolerance criteria are not achieved, corrective action specified in the balance calibration SOP must be applied before the balance can be used for laboratory measurements. All weights are recalibrated by outside vendor every five years following the specifications for weight calibration verification detailed in the United States Environmental Protection Agency's "Manual for the Certification of Laboratories Analyzing Drinking Water", Fifth Edition, January 2005. Certificate(s) of calibration are maintained on file with QAO. Balances are inspected and maintained by professional service technicians annually. Certificate(s) of inspection are maintained with QAO.

7.3 ***Traceability of Chemical Standards and Reagents***. All chemicals and reagents, purchased as reference standards for use in method calibration must establish traceability to NIST referenced material through a traceability certificate (Certificate of Analysis, CoA). Process links are established that enable a calibration standard

solution to be traced to its NIST reference certificate. Solvents, acids and other supplies are being tested to verify their suitability for the analytical process.

7.4 ***Assignment Of Reagent and Standard Expiration Dates.*** Expiration date information for all purchased standards and reagents is provided to SGS Accutest – Orlando with all prepared standard solutions and unstable reagents as a condition of purchase. Neat materials and inorganic reagents are not required to be purchased with expiration dates. Certified prepared solutions are labeled with the expiration date provided by the manufacturer. In-house prepared solutions are assigned expiration dates that are consistent with the method that employs their use unless documented experience indicates that an alternate date can be applied. If alternate expiration dates are employed, their use is documented in the method SOP. Expiration dates for prepared inorganic reagents, which have not exhibited instability, are established at two years form the date of preparation for tracking purposes. All containers shall be labeled with the date of preparation and expiration date clearly indicated.

The earliest expiration date is always the limiting date for assigning expiration dates to prepared solutions. Expiration dates that are later than the expiration date of any derivative solution or material are prohibited.

7.5 ***Documentation of Traceability***. Traceability information is documented in individual logbooks designated for the measurement process in use. The QA Officer maintains calibration documentation for metric references in pertinent folders and logbooks.

Balance calibration verification is documented in logbooks that are assigned to each balance. The individual conducting the verification is required to initial and date all calibration activities. Any defects that occur during verification are also documented along with the corrective action applied and a demonstration of return to control. Annual service and calibration reports and certificates retained on file with QA staff.

Temperature control is documented in logbooks assigned to the equipment being monitored. A verified (see 7.1) thermometer is assigned to each individual item. Measurements are recorded along with date and initials of the individual conducting the measurement on a daily or as used basis. Corrective action, if required, is also documented including the demonstration of return to control.

Initial traceability of chemical standards and reagents is documented via a vendor-supplied certificate (see also 7.3) that includes lot number and expiration date information. Solutions prepared using the vendor supplied chemical standards are documented in logbooks assigned to specific analytical processes. Alternatively, documentation may be entered into the electronic standards and reagent tracking log The documentation includes links to the vendors lot number, an internal lot number, dates of preparation, and the preparer's initials. Standards received without certificate of analysis can not be used for calibration or calibration verification and are rejected.

Supervisors conduct regular reviews of logbooks, which are verified using a word rev'd", signature and date. QA Staff monitors the process and documents it in the same manner.

8.0 TEST PROCEDURES, METHOD REFERENCES, AND REGULATORY PROGRAMS

Requirements: The laboratory must use client specified or regulatory agency approved methods for the analysis of environmental samples. The laboratory maintains a list of active methods, which specifies the type of analysis performed, and cross-references the methods to applicable environmental regulation. Routine procedures used by the laboratory for the execution of a method must be documented in a standard operating procedure. Method performance and sensitivity must be demonstrated annually where required. Defined procedures for the use of method sensitivity for data reporting purposes must be established by the Director of Quality Assurance and used consistently for all data reporting purposes.

8.1 ***Method Selection.*** SGS Accutest – Orlando employs methods for environmental sample analysis that are consistent with the client's application, which are appropriate and applicable to the project objectives. SGS Accutest – Orlando informs the client if the method proposed is inappropriate or outdated and suggests alternative approaches.

SGS Accutest – Orlando employs documented, validated regulatory methods in the absence of a client specification and informs the client of the method selected. These methods are available to the client and other parties as determined by the client. Documented and validated in-house methods may be applied if they are appropriate to the project. The client is informed of the method selection.

8.2 ***Method Validation***. Standard methods from regulatory sources are primarily used for all analysis. Standard methods do not require validation by the laboratory. Non-standard, in-house methods are validated prior to use. Validation is also performed for standard methods applied outside their intended scope of use. Validation is dependent upon the method application and may include analysis of quality control samples to develop precision and accuracy information for the intended use. A final method validation report is generated, which includes all data in the validation study. A statement of adequacy and/or equivalency is included in the report. A copy of the report is archived in the quality assurance directory of the company server.

Non-standard methods are validated prior to use. This includes the validation of modified standard methods to demonstrate comparability with existing methods. Demonstrations and validations are performed and documented prior to incorporating technological enhancements and non-standard methods into existing laboratory methods used for general applications. The demonstration includes method specific requirements for assuring that significant performance differences do not occur when the enhancement is incorporated into the method. Validation is dependent upon method application and may include the analysis of quality control samples to develop precision and accuracy information for intended use.

The study procedures and specifications for demonstrating validation include comparable method sensitivity, calibration response, method precision, method accuracy and field sample consistency for several classes of analytical methods are

detailed in this document. These procedures and specifications may vary depending upon the method and the modification.

8.3 ***Standard Operating Procedures***. Standard operating procedures (SOP) are prepared for routine methods executed by the laboratory and processes related to sample or data handling. The procedures describe the process steps in sufficient detail to enable an individual, who is unfamiliar with the procedure to execute it successfully. SOPs are reviewed annually and edited if necessary. SOPs can be edited on a more frequent basis if systematic errors dictate a need for process change or the originating regulatory agency promulgates a new version of the method. Procedural modifications are indicated using a revision number. SOPs are available for client review at the SGS Accutest – Orlando facility upon request.

8.4 ***Method Detection Limit Determination and verification***. Annual method detection limit (MDL) studies are performed as appropriate for routine methods used in the laboratory. MDL studies are also performed when there is a change to the method that affects how the method is performed or when an instrumentation change that impacts sensitivity occurs. The procedure used for determining MDLs is described in 40 CFR, Part 136, Appendix B. Studies are performed for each method on water, soil and air matrices for every instrument that is used to perform the method. MDLs are established at the instrument level. The highest MDL of the pooled instrument data is used to establish a laboratory MDL. MDLs are experimentally verified through the analysis of spiked quality control samples at 2-3 times the concentration of the experimental MDL, or 1-4 times for multicomponent methods. The verification is performed on every instrument used to perform the analysis. The quality assurance staff manages the annual MDL determination process and is responsible for retaining MDL data on file. Approved MDLs are appended to the LIMS and used for data reporting purposes. MDL values are used as DL values for DOD projects and verification spiking concentrations are listed as LOD values.

Methods certified under DOD ELAP requirements must undergo verification procedure on quarterly basis – see DOD QSM 5.0,Volume 1, Module 4, 1.5.2.1.g)

8.5 ***Method Reporting Limit.*** The method reporting limit is established at the lowest concentration calibration standard in the calibration curve. The low calibration standard is selected by department managers as the lowest concentration standard that can be used while continuing to meet the calibration linearity criteria of the method being used. The validity of the Method Reporting Limits is confirmed via analysis of a spiked quality control sample at 1 – 2x Method reporting limit concentration. RL values are referred to as LOQ for DOD projects.

By definition, detected analytes at concentrations below the low calibration standard cannot be accurately quantitated and must be qualified accordingly.

Methods certified under DOD ELAP requirements must undergo verification procedure on quarterly basis – see DOD QSM 5.0,Volume 1, Module 4, 1.5.2.2.e).

8.6 ***Reporting of Quantitative Data.*** Analytical data for all methods is reported without qualification to the reporting limit established for each method. Data may be reported to MDL depending upon the client's requirements provided that all qualitative identification criteria for the parameter have been satisfied. All parameters reported at concentrations between the reporting limit and MDL are qualified as an estimated concentration.

Measured concentrations of detected analytes that exceed the upper limit of the calibration range are either diluted into the range and reanalyzed or qualified as an estimated value. The only exception to this applies to ICP and ICP/MS analysis, which can be reported to the upper limit of the experimentally determined linear range without qualification.

8.7 ***Estimated Uncertainty.*** A statement of the estimated uncertainty of an analytical measurement accompanies the test result when required. Estimated uncertainty is derived from the performance limits established for spiked samples of similar matrices. The degree of uncertainty is derived from the negative or positive bias for spiked samples accompanying a specific parameter. When the uncertainty estimate is applied to a measured value, the possible quantitative range for that specific parameter at that measured concentration is defined. Well recognized regulatory methods that specify values for the major sources of uncertainty and specify the data reporting format do not require a further estimate of uncertainty.

8.8 ***Precision and Accuracy Studies***. Annual precision and accuracy (P&A) studies, which demonstrate the laboratories ability to generate acceptable date, are performed for all routine methods used in the laboratory. The procedure used for generating P&A data is referenced in the majority of the regulatory methodology in use. The procedure requires quadruplicate analysis of a sample spiked with target analytes at a concentration in the working range of the method. This data may be compiled from a series of existing blank spikes or laboratory control samples. Accuracy (percent recovery) of the replicate analysis is averaged and compared to established method performance limits. Values within method limits indicate an acceptable performance demonstration. (See also Sec 4, Training, Demonstration of capability)

8.9 ***Method Sources, References and Update Mechanism.*** The Quality Assurance Staff maintains a list of active methods used for the analysis of samples. This list includes valid method references such as EPA, American Society of Testing and Materials (ASTM) or Standard Methods designations and the current version and version date.

Updated versions of approved reference methodology are placed into use as changes occur. The Quality Assurance Director informs operations management of changes in method versions as they occur. The operations management staff selects an implementation date. The operations staff is responsible for completing all method requirements prior to the implementation date. This includes modification to SOPs, completion of MDL and precision and accuracy studies and staff training. Documentation of these activities is provided to the QA staff who retains this information on file. The updated method is placed into service on the implementation date and the old version is de-activated.

Multiple versions of selected methods may remain in use to satisfy client specific needs. In these situations, the default method version becomes the most recent version. Client specific needs are communicated to the laboratory staff using method specific analytical codes, which clearly depict the version to be used. The old method version is maintained as an active method until the specified client no longer requires the use of the older version.

SGS Accutest – Orlando will not use methodology that represents significant departures from the reference method unless specifically directed by the client. In cases where clients direct the laboratory to use a method modification that represents a significant departure from the reference method, the request will be documented in the project file. The LQSM lists active methods used for the analysis of samples in Table 8.1. This list includes valid method references from sources such as USEPA, ASTM or Standard Methods designations and the current version and version date.

8.10 ***Analytical Capabilities***. Appendix II provides a detailed listing of the methodology employed for the analysis of test samples.

9.0 SAMPLE MANAGEMENT, LOGIN, CUSTODY, STORAGE AND DISPOSAL

Requirement: A system to ensure that client supplied product is adequately evaluated, acknowledged, and secured upon delivery to the laboratory must be practiced by the laboratory. The system must assure that chain of custody is maintained and that sample receipt conditions and preservation status are documented and communicated to the client and internal staff. The login procedure must assign, document, and map the specifications for the analysis of each unique sample to assure that the requested analysis is performed on the correct sample and enables the sample to be tracked throughout the laboratory analytical cycle. The system must include procedures for reconciling defects in sample condition or client provided data, which occur at sample arrival. The system must specify the procedures for proper sample storage, transfer to the laboratory, and disposal after analysis. The system must be documented in a standard operating procedure.

9.1 ***Order Receipt and Entry***. New orders are initiated and processed by the client services group (See Chapter 14, Procedures for Executing Client Specifications). The new order procedure includes mechanisms for providing sampling containers to clients. These containers must meet the size, cleanliness, and preservation specifications for the analysis to be performed.

For new orders, the project manager prepares a bottle request form, which is submitted to sample management department. This form provides critical project details to the sample management staff, which are used to prepare and assemble the sample bottles for shipment to the client prior to sampling.

The bottle order is assembled using bottles that meet USEPA specifications for contaminant-free sample containers. SGS Accutest – Orlando checks all sample containers for cleanliness. Data are reviewed by both the analyst and sample management technician. Results of bottle analyses are retained for minimum of 5 years.

All preservative solutions are prepared in the laboratory and are checked to assure that they are free of contamination from analytes of interest before being released for use. Sample management department retains a copy of the documentation of in-house contamination checks.

Reagent water for trip and field blanks is poured into appropriately labeled containers. Sample bottleware is labeled with durable labels printed on waterproof printing medium with indelible laser or heat transfer printer ink. All bottles are packed into ice chests with blank chain of custody forms and the original bottle order form. Completed bottle orders are delivered to clients using SGS Accutest – Orlando couriers or commercial carriers for use in field sample collection.

9.2 ***Sample Receipt and Custody***. Samples are delivered to the laboratory using a variety of mechanisms including SGS Accutest – Orlando couriers, commercial shippers, and client self-delivery. Documented procedures are followed for arriving

samples to assure that custody and integrity are maintained and that handling and preservation requirements are documented and continued.

Sample custody documentation is initiated when the individual collecting the sample collects field samples. Custody documentation includes all information necessary to provide an unambiguous record of sample collection, sample identification, and sample collection chronology. Initial custody documentation employs either SGS Accutest – Orlando or client generated custody forms.

SGS Accutest – Orlando generates a Sample Receipt Confirmation form in situations where the individuals who collected the sample did not generate custody documentation in the field. SGS Accutest – Orlando Project Manager then contacts the client for the CoC information to be faxed or e-mailed from the client to the lab.

SGS Accutest – Orlando defines sample custody as follows:

- The sample is in the actual custody or possession of the assigned responsible person,

- The sample is in a secure area.

The SGS Accutest – Orlando facility is defined as a secure facility. Perimeter security has been established, which limits access to authorized individuals only. Visitors enter the facility through the building lobby and must register with the receptionist prior to entering controlled areas. While in the facility, visitors must be accompanied by their hosts at all times. After hours, building access is controlled using a computerized pass-key reader system. This system limits building access to individuals with a pre-assigned authorization status. After hours visitors are not authorized to be in the building. Clients delivering samples after hours must make advanced arrangements through client services and sample management to assure that staff is available to take delivery and maintain custody.

Upon arrival at SGS Accutest – Orlando, the sample custodian reviews the chain of custody and generates Sample Receipt Confirmation form for the samples received to verify that the information on the form corresponds with the samples delivered. This includes verification that all listed samples are present and properly labeled, checks to verify that samples were transported and received at the required temperature, verification that the sample was received in proper containers, verification that sufficient volume is available to conduct the requested analysis, and a check of individual sample containers to verify test specific preservation requirements including the absence of headspace for volatile compound analysis.

9.3 Sample conditions and other observations are documented on the Sample Receipt Confirmation form by the sample custodian prior to completing acceptance of custody. The sample custodian accepts sample custody upon verification that the custody document is correct. Discrepancies or non-compliant situations are documented, flagged and communicated to the SGS Accutest – Orlando project manager, who

contacts the client for resolution. The resolution is documented and communicated to sample management for execution.

9.4 ***Laboratory preservation of Improperly preserved field samples.*** SGS Accutest – Orlando extends every effort to preserve samples which were received without proper field preservation.

Field/Equipment negative controls also receive the same amount of preservation as incorrectly preserved samples, and record made in the preservation logbook.

9.5 ***Sample Tracking Via Status Change.*** An automated, electronic LIMS procedure records sample exchange transactions between departments and changes in analytical status. This system tracks all preparation, analytical, and data reporting procedures to which a sample is subjected while in the possession of the laboratory. Each individual receiving samples must acknowledge the change in custody and operational status in the LIMS. This step is required to maintain an accurate electronic record of sample status, dates of analytical activity, and custody throughout the laboratory.

Sample tracking is initiated at login where all chronological information related to sample collection dates and holding times are entered into the LIMS. This information is entered on an individual sample basis

9.6 ***Sample Acceptance Policy***. Incoming samples must satisfy SGS Accutest – Orlando sample acceptance criteria before being logged into the system. Sample acceptance is based on the premise that clients have exercised proper protocols for sample collection. This includes sufficient volume, proper chemical preservation, temperature preservation, sample container sealing and labeling, and appropriate shipping container packing.

The sample management staff will make every attempt to preserve improperly preserved samples upon arrival. However, if preservation is not possible, the samples may be refused unless the client authorizes analysis. No samples will be accepted if holding times have been exceeded or will be exceeded before analysis can take place unless the client authorizes analysis.

Sample acceptance criteria include proper custody and sample labeling documentation. Proper custody documentation includes an entry for all physical samples delivered to the laboratory with an identification code that matches the sample bottle and a date and signature of the individual who collected the sample and delivered them to the laboratory. Labeling is done using durable waterproof labels printed with indelible heat-transfer ink.

SGS Accutest – Orlando reserves the right to refuse any sample which in its sole and absolute discretion and judgement is hazardous, toxic and poses or may pose a health, safety or environmental risk during handling or processing. The company will not accept samples for analysis using methodology that is not performed by the laboratory or for

methods that lab does not hold valid accreditation unless arrangements have been made to have the analysis conducted by a qualified subcontractor.

9.7 ***Assignment of Unique Sample Identification Codes***. Unique identification codes must be assigned to each sample bottle to assure traceability and unambiguously identify the tests to be performed in the laboratory.

The sample identification coding process begins with the assignment of a unique alphanumeric job number. A job is defined as a group of samples received on the same day, from a specific client pertaining to a specific project. A job may consist of groups of samples received over multi-day period. The first character of the job number is an alpha-character that identifies the laboratory facility. The next characters are numeric and sequence by one number with each new job.

Unique sample numbers are assigned to each bottle collected as a discrete entity from a designated sample point. This number begins with the job number and incorporates a second series of numbers beginning at one and continuing chronologically for each point of collection. The test to be performed is clearly identified on the bottle label.

Alpha suffixes may be added to the sample number to identify special designations such as subcontracted tests, in-house QC checks, or re-logs. Multiple sample bottles for a specific analysis are labeled Bottle 1, Bottle 2, etc.

9.8 ***Subcontracted Analysis***. Subcontract laboratories are employed to perform analysis not performed by SGS Accutest - Orlando. The quality assurance staff evaluates subcontract laboratories to assure their quality processes meet the standards of the environmental laboratory industry prior to engagement. Throughout the subcontract process, SGS Accutest – Orlando follows established procedures to assure that sample custody is maintained and the data produced by the subcontractor meets established quality criteria.

SGS Accutest network laboratories are considered primary subcontractors.

*Subcontracting Procedure***.** Subcontracting procedures are initiated through several mechanisms, which originate with sample management. Samples for analysis by a subcontractor are logged into the SGS Accutest – Orlando system using regular login procedures. If subcontract parameters are part of the project or sample management has received subcontracting instructions for a specific project, a copy of the chain of custody is given to the appropriate project manager with the subcontracted parameters highlighted. This procedure triggers the subcontract process at the project management level. The Sample Management supervisor contacts an approved subcontractor to place the subcontract order. Subcontract chain of custody is processed in Sample Management Department and copy is filed with the original CoC. Sample management signs the subcontract chain of custody and ships the sample(s) to the subcontractor. The subcontract COC is filed with the original COC and the request for subcontract. Copies are distributed to the login department, the project manager, and sample management.

Client is verbally notified by Project Manager of the requirement to subcontract to the outside laboratory as soon as need is identified by the SGS Accutest – Orlando staff. Client notification must be verified in writing, i.e. by e-mail. Client notification may take place during the initial project set-up, or at the time of sample receipt and login.

Subcontractor data packages are reviewed by the QA Staff to assess completeness and quality compliance. If completeness defects are detected, the subcontractor is asked to immediately upgrade the data package. If data quality defects are detected, the package is forwarded to the QA staff for further review. The QA staff will pursue a corrective action solution before releasing data to the client.

Approved subcontract data is entered into the laboratory information management system (LIMS) if possible and incorporated into the final report. All subcontract data is footnoted to provide the client with a clear indication of its source. Copies of original subcontract data are always included in the data report whether in hardcopy or PDF file, depending on the data submission requirements.

*Subcontract Laboratory Evaluation***.** The QA staff evaluates subcontract laboratories prior to engagement. As a minimum, the subcontract laboratory must provide SGS Accutest – Orlando with proof of a valid certification to perform the requested analysis for the venue where they were collected, QC criteria summary (LOD/LOQ, LCS, MS/MSD, %RPD, etc.), copy of the most recent regulatory agency audit report, and a copy of the laboratory's Summary of Qualifications (SOQ). Other beneficial materials are QSM, copies of SOPs used for the subcontracted analysis, a copy of the most recent performance evaluation study for the subcontracted parameter, and copies of the most recent third party accreditor's audit report.

Certification verification must be submitted to SGS Accutest – Orlando annually. If possible, the QA staff may conduct a site visit to the laboratory to inspect the quality system. SGS Accutest - Orlando assumes the responsibility for the performance of all subcontractors who have successfully demonstrated their qualifications. When selecting a subcontractor for analysis not performed by SGS Accutest – Orlando, assure qualifications of the subcontractor through local QA officer.

Qualification process of a subcontract laboratory may be bypassed if the primary client directs SGS Accutest – Orlando to employ a specific subcontractor

Subcontract Laboratory Database. SGS Accutest Inc. maintains centralized database of preferred contractors in order to optimize sample handling and data submission process, as well as obtain competitive priced services of uniform quality throughout the network. Individual SGS Accutest facilities are assigned "Center of Expertise" status according to unique capabilities.

9.9 ***Sample Storage***. Following sample custody transfer, samples are assigned to various refrigerated storage areas by the sample management staff depending upon the test to be performed and the matrix of the samples. The location (refrigerator and shelf) of each sample is entered into sample location database on the line

corresponding to each sample number. Samples remain in storage until the laboratory technician retrieves them into the laboratory for analysis.

Samples for volatile organics analysis are placed in storage in designated refrigerators by the sample management staff and immediately transferred to the organics group control. Sample custody is transferred to the VOC department staff. These samples are segregated according to matrix to limit opportunities for cross contamination to occur.

Organics staff is authorized to retrieve samples from these storage areas for analysis. When analysis is complete, the samples are placed back into storage.

9.10 ***Sample Login***. Following sample custody transfer to the laboratory, the documentation that describes the clients analytical requirements are delivered to the sample login group for coding and entry to the Laboratory Information management System (LIMS). This process translates all information related to collection time, turnaround time, sample analysis, and deliverables into a code which enables client requirements to be electronically distributed to the various departments within the laboratory for scheduling and execution.

The technical staff is alerted to client or project specific requirements through the use of a unique project code that is electronically attached to the job during login. The unique project code directs the technical staff to controlled specifications documents detailing the unique requirements.

9.11 ***Sample Retrieval for Analysis***. It is a responsibility of individual analyst to retrieve samples for analysis. Sample Management employs a program to facilitate sample placement and retrieval. Sample is traced around the laboratory using Status feature of LIMS.

After sample analysis has been completed, the analyst places the sample back into the storage and updates sample status.

9.12 ***Sample Disposal***. SGS Accutest - Orlando retains all samples under proper storage for a minimum of 30 days following completion of the analysis report. Longer storage periods are accommodated on a client specific basis if required. Samples may also be returned to the client for disposal.

SGS Accutest – Orlando disposes of all laboratory wastes following the requirements of the Resource Conservation and Recovery Act (RCRA). The Company has obtained and maintains a waste generator identification number, FLR00001263309002 (FLR designates State of Florida).

Sample management generates a sample disposal dump sheet from the LIMS tracking system each week, which lists all samples whose holding period has expired. Data from each sample is compared to the hazardous waste criteria established by the Florida Department of Environmental Protection (FDEP).

Samples containing constituents at concentrations above the criteria are labeled as hazardous and segregated into the following waste categories for disposal as follows:

Chlorinated Waste (Closed Top Steel Drum)- Methylene Chloride

Non-Chlorinated Waste (Closed Top Steel Drum)- Hexane, Methanol, and mixed solvents

Sodium Sulfate/Used Charcoal (Open Top Steel Drum)- Charcoal and paper filters used in the filtering of samples.

Hazardous Flammable Vials (Open Top Polypropylene Drum)- Methylene Chloride, Hexane.

Hazardous Aqueous waste (Closed Top Polypropylene Drum)- High Odor Samples, Lachat Waste.

Non Hazardous Soil (Open Top Steel Drum)- Soils.

Hazardous Solid Waste- (Open Top Steel Drum).

Non-Aqueous/Oil Samples- (Closed Top Steel Drum)

Difference between Open and Closed type of drums is whether it is possible to remove entire lid or just threaded stopper. Drums are closed at all times while in storage.

Non-hazardous aqueous samples are neutralized and collected in HDPP 500 Gal holding tank to be removed by waste company.

Non-hazardous solids are drummed and disposed of by contract waste company. Sample bottles are disposed of as recyclable waste in order to crush the bottles and destroy the labels. VOC vials are crushed on site using PRODEVA glass crusher. Supernatant liquid is siphoned off into the HDPP holding tank and solid residue drummed separately.

Laboratory wastes are collected by waste stream in designated areas throughout the laboratory. Waste streams are consolidated twice a week by the waste custodian and transferred to stream specific drums for disposal through a permitted waste management contractor. Filled, consolidated drums are tested for hazardous characteristics and scheduled for removal from the facility for appropriate disposal based on the laboratory data.

10.0 LABORATORY INSTRUMENTATION AND MEASUREMENT STANDARDS

Requirement: Procedures, which assure that instrumentation is performing to a pre-determined operational standard prior to the analysis of any samples, must be established by the laboratory. In general, these procedures will follow the regulatory agency requirements established in promulgated methodology. The instrumentation selected to perform specified analysis is capable of providing the method-specified uncertainty and sufficient sensitivity of measurement needed. These procedures must be documented and incorporated into the standard operating procedures for the method being executed. SASE Equipment List attached as Appendix III.

10.1 ***Mass Tuning – Mass Spectrometers***. The mass spectrometer tune and sensitivity must be monitored to assure that the instrument is assigning masses and mass abundances correctly and that the instrument has sufficient sensitivity to detect compounds at low concentrations. This is accomplished by analyzing a specific mass tuning compound at a fixed concentration. If the sensitivity is insufficient to detect the tuning compound, corrective action must be performed prior to the analysis of standards or samples. If the mass assignments or mass abundances do not meet criteria, corrective action must be performed prior to the analysis of standards or samples.

10.2 ***Wavelength Verification – Spectrophotometers***. Spectrophotometer detectors are checked on a regular schedule to verify proper response to the wavelength of light needed for the test in use. If the detector response does not meet specifications, corrective action (detector adjustment or replacement) is performed prior to the analysis of standards or samples.

10.3 ***Inter-element Interference Checks (Metals)***. Inductively Coupled Plasma Emission Spectrophotometers (ICP) are subject to a variety of spectral interferences, which can be minimized or eliminated by applying interfering element correction factors and background correction points. Interfering element correction factors are checked on a specified frequency through the analysis of check samples containing high levels of interfering elements. Analysis of single element interferent solutions is also conducted at a specified frequency.

If the check indicates that the method criteria has not been achieved for any element in the check standard, the analysis is halted and data from the affected samples are not reported. Sample analysis is resumed after corrective action has been performed and the correction factors have been re-calculated.

New interfering element correction factors are calculated and applied whenever the checks indicate that the correction factors are no longer meeting criteria. At a minimum, correction factors are replaced once a year.

10.4 ***Calibration and Calibration Verification***. Many tests require calibration using a series of reference standards to establish the concentration range for performing quantitative analysis. Method specific procedures for calibration are followed prior to any sample analysis.

Calibration is performed using a linear or quadratic regression calculation or calibration factors calculated from the curve. The calibration must meet method specific criteria for linearity or precision. If the criteria are not achieved, corrective action (instrument maintenance or re-calibration) is performed. The instrument must be successfully calibrated before analysis of samples can be conducted.

Initial calibration for metals analysis performed using inductively coupled plasma (ICP) employs the use of two standards and a calibration blank to establish linearity. The calibration blank contains all reagents that are placed into the calibration standard with the exception of the target elements. Valid calibration blanks must not contain any target elements.

Initial calibrations must be initially verified using a single concentration calibration standard from a second source (i.e. separate lot or different provider). The continuing validity of an existing calibration must be regularly verified using a single concentration calibration standard. The response to the standard must meet pre-established criteria that indicate the initial calibration curve remains valid. Samples must be bracketed by passing CCVs. If the criteria are not achieved corrective action (re-calibration) is performed before any additional samples may be analyzed.

10.5 ***Linear Range Verification and Calibration*** Linear range verification is performed for all ICP, ICP/MS and select General Chemistry methods. The regulatory program or analytical method specifies the verification frequency. A series of calibration standards are analyzed over a broad concentration range. The data from these analyses are used to determine the valid analytical range for the instrument.

Some methods or analytical programs require a low concentration calibration check to verify that instrument is sufficient to detect target elements at the reporting limit. The analytical method or regulatory program defines the criteria used to evaluate the low concentration calibration check. If the low calibration check fails criteria, corrective action is performed and verified through reanalysis of the low concentration calibration check before continuing with the field sample analysis.

In accordance with TNI standards minimum number of calibration points in the absence of method-specific requirements is two calibration points and a blank.

10.6 ***Retention Time Verification (GC/HPLC/IC)***. Chromatographic retention time windows are developed for all analysis performed using gas chromatographs with conventional detectors. An initial experimental study is performed, which establishes the width of the retention window for each compound. The retention time range of the window defines the time ranges for elution of specified target analytes on the primary and

confirmation columns. Retention time windows are established upon initial calibration, applying the retention time range from the initial study to each target compound. Retention times are regularly confirmed through the analysis of an authentic standard during calibration verification. If the target analytes do not elute within the defined range during calibration verification, the instrument must be recalibrated and new windows defined. New studies are performed when major changes, such as column replacement are made to the chromatographic system.

11.0 INSTRUMENT MAINTENANCE

***Requirement*.** Procedures must be established for equipment maintenance. The procedure may include a maintenance schedule if required or documentation of daily maintenance related activities. All instrument maintenance activities must be documented in instrument specific logbooks. All equipment out of service (both analytical and auxiliary) must be clearly marked "Out of Order".

11.1 ***Routine, Daily Maintenance***. Routine, daily maintenance is required on an instrument specific basis. It is performed each time the instrument is used. Daily maintenance traditionally includes activities to insure a continuation of good analytical performance. In some cases, they include performance checks that indicate whether non-routine maintenance is required. If the performance check indicates a need for higher level maintenance, the equipment is taken out of service until maintenance is performed. Analysis cannot be continued until the performance checks meet established criteria. Document return to control. Daily maintenance is the responsibility of the individual assigned to the instrument used for the analysis he is performing.

11.2 ***Non-routine Maintenance***. Non-routine maintenance is reserved for catastrophic occurrences such as instrument failure. The need for non-routine maintenance is indicated by failures in general operating systems that result in an inability to conduct required performance checks or calibration. Equipment in this category are taken out of service and repaired before attempting further analysis. Analysis cannot continue until the instrument meets all performance check criteria and is capable of being calibrated. Section supervisors are responsible for identifying non-routine maintenance episodes and initiating repair activities to bring the equipment on-line. This may include initiating telephone calls to maintenance contractors if necessary. They are also responsible for documenting all details related to the occurrence and the repair.

11.3 ***Scheduled Maintenance***. Modern laboratory instrumentation rarely requires traditional scheduled maintenance. Where required, the equipment is placed on a schedule, which dictates when maintenance is required. Examples include annual balance calibration by an independent provider and pump oil changes. Section supervisors are responsible for initiating scheduled maintenance on equipment that requires scheduled preventative attention. Scheduled maintenance is documented using routine documentation practices.

11.4 ***Maintenance Documentation***. Routine and non-routine maintenance activities are documented in logbooks assigned to instruments and equipment used for analytical measurements. The logbooks contain preprinted forms, which specify the maintenance activities required with each use. SGS Accutest - Orlando has adopted a problem – action – follow-up format to conduct instrument maintenance. The analyst or supervisor who performs or initiates the maintenance activity is required to check the activity upon its completion, verify complete statement of return to normal conditions and initial the form. Non-routine maintenance (i.e. repairs, upgrades, etc.) is documented as well either electronically via e-mail from the service provider or receipt attached to the maintenance log.

12.0 QUALITY CONTROL PARAMETERS, PROCEDURES, AND CORRECTIVE ACTION

Requirement: All procedures used for test methods must incorporate quality control parameters to monitor elements that are critical to method performance. Each quality parameter includes acceptance criteria that have been established by regulatory agencies for the methods in use. Criteria may also be established through client dictates or through the accumulation and statistical evaluation of internal performance data. Data obtained from these parameters must be evaluated by the analyst, and compared to established method criteria. If the criteria are not achieved, the procedures must specify corrective action and conformation of control before proceeding with sample analysis. QC parameters, procedures, and corrective action must be documented within the standard operating procedures for each method. In the absence of client specific objectives the laboratory must define qualitative objectives for completeness and representativeness of data.

12.1 ***Procedure***. Bench analysts are responsible for methodological quality control and sample specific quality control. Each method specifies the control parameters to be employed for the method in use and the specific procedures for incorporating them into the analysis. These control parameters are analyzed and evaluated with every designated sample group (batch).

The data from each parameter provides the analyst with critical decision making information on method performance. The information is used to determine if corrective action is needed to bring the method or the analysis of a specific sample into compliance. These evaluations are conducted throughout the course of the analysis. Each parameter being indicative of a critical control feature. Failure of a methodological control parameter is indicative of either instrument or batch failure. Failure of a sample control parameter is indicative of control difficulties with a specific sample or samples.

Sample Batch. All samples analyzed in the laboratory are assigned to a designated sample batch, which contains all required quality control samples and a defined maximum number of field samples that are prepared and/or analyzed over a defined time period. The maximum number of investigative and field QC samples in the batch is 20. SGS Accutest – Orlando has incorporated the TNI batching policy as the sample-batching standard. This policy incorporates the requirement for blanks and spiked blanks as a time based function as defined by TNI. The typical batch contains a blank, laboratory control sample (LCS or spiked blank), matrix spike and matrix spike duplicate. Batch documentation includes lot specifications for all reagents and standards used during preparation of the batch.

12.2 ***Methodological Control Parameters and Corrective Action***. Prior to the analysis of field sample the analyst must determine that the method is functioning properly. Specific control parameters indicate whether critical processes meet specified requirements before continuing with the analysis. Method specific control parameters must meet criteria before sample analysis can be conducted. Each of these

parameters is related to processes that are under the control of the laboratory and can be adjusted if out of control.

Method Blank. A method blank is analyzed during the analysis of any field sample. The method blank is defined as a sample. It contains the same standards (internal standards, surrogates, matrix modifiers, etc.) and reagents that are added to the field sample during analysis, with the exception of the sample itself. If the method blank contains target analyte(s) at concentrations that exceed method or client requirements (typically defined as 1/2 RL concentrations), the source of contamination is eliminated before proceeding with sample analysis. Systematic contamination is documented for corrective action and resolved following the established corrective action procedures. In specific cases, contamination detected in the method blank may be acceptable if the concentrations do not exceed regulatory limits or client defined reporting limits.

Laboratory Control Samples (LCS or Spiked Blanks). A laboratory control sample (spiked blank or commercially prepared performance evaluation sample) is analyzed along with field samples to demonstrate that the method accuracy is within acceptable limits. These spike solutions are derived from different sources than the solutions used for method calibration. The performance limits are derived from published method specifications or from statistical controls generated from laboratory method performance data. Spiked blanks are blank matrices (reagent water, clean sand, Teflon chips, or granular sodium sulfate) spiked with the targeted parameters and analyzed using the same method used for samples. Accuracy data is compared to laboratory experimentally derived limits to determine if the method is in control. Laboratory control samples (LCS) may be laboratory or commercially prepared spiked samples in an inert material.

Accuracy data is compared to the applicable performance limits. If the spike accuracy exceeds the performance limits, corrective action, as specified in the SOP for the method is performed and verified before continuing with a field sample analysis. In some cases, decisions are made to continue with sample analysis if performance limits are exceeded; provided the unacceptable result has no negative impact on the sample data.

Marginal exceedance (ME) values are calculated for methods containing more than eleven (11) targeted analytes. The ME is calculated as $\pm$ 4 standard deviations about the mean. MEs are considered for multi-analyte methods because of the increased likelihood of LCS failure as the number of analytes in the method increase. The number of allowable MEs is based on the number of target analytes in the method. Analytes that regularly fall into the ME category are treated as systematic problems, which are resolved using established trend monitoring and corrective action procedures. Marginal Exceedances are not applied to parameters that are detected in field samples. Routine corrective action is initiated for all cases where LCS spike accuracy criteria is beyond the established control limits and the parameter is detected in field samples corresponding to the unacceptable LCS. Use of ME may be disallowed on project-specific basis.

Blanks and spikes are routinely evaluated before samples are analyzed. However, in situations where sample analysis is performed using an autosampler, they may be evaluated after sample analysis has occurred. If the blanks and spikes do not meet criteria, sample analysis is repeated.

Proficiency Testing. Performance Evaluation (Proficiency Testing) samples (PEs, PTs) are single or double blind samples spiked with know amount of analytes on interest and introduced to the laboratory to assess method performance. PEs may be introduced as double blinds submitted by commercial clients, single or double blinds from regulatory agencies, or internal blinds submitted by the QA group.

A minimum of two single blind studies must be performed each year for every parameter in aqueous and solid matrices for each field of proficiency testing (FOPT) for which the laboratory maintains accreditation. Proficiency Testing samples must be purchased as blinds from an accredited vendor for every combination of analyte-matrix-method. Data from these studies are provided to the laboratory by the vendor and reported to accrediting agencies. If unsatisfactory performance is noted, corrective action is performed to identify and eliminate any sources of error. A new PT must be analyzed to demonstrate continuing proficiency.

PE samples performed for accrediting agencies or clients, which do not meet performance specifications, require a written summary that documents the corrective action investigation, findings, and corrective action implementation.

Single or double blind PT samples are employed for self-evaluation purposes. Data from these analyses are compared to established performance limits. If the data does not meet performance specifications, the system is evaluated for sources of acute or systematic error. If required, corrective action is performed and verified before initiating or continuing sample analysis.

Trend Analysis for Control Parameters. Accuracy data for selected spiked parameters from the laboratory control sample (LCS) is statistically evaluated daily for trends. Data from selected LCS parameters and surrogates are pooled on a method, matrix, and instrument basis. This data is evaluated by comparison to existing control and warning limits. Trend analysis is performed automatically as follows:

- Any point outside the control limit
- Any three consecutive points between the warning and control limits
- Any eight consecutive points on the same side of the mean
- Any six consecutive points increasing or decreasing

The results of the trend analysis are printed for supervisory evaluation prior to sample analysis. Trends that indicate the potential loss of statistical control are further evaluated to determine the impact on data quality and to determine if corrective action is necessary. If corrective action is indicated, the supervisor informs the analysts of

the corrective actions to be performed. Return to control is demonstrated before analysis resumes.

12.3 ***Sample Control Parameters and Corrective Action***. The analysis of samples can be initiated following a successful demonstration that the method is operating within established controls. Additional controls are incorporated into the analysis of each sample to determine if the method is functioning within established specifications for each individual sample. Sample QC data is evaluated and compared to established performance criteria. If the criteria are not achieved the method or the SOP specifies the corrective action required to continue sample analysis. In many cases, failure to meet QC criteria is a function of sample matrix and cannot be remedied. Each parameter is designed to provide quality feedback on a defined aspect of the sampling and analysis episode.

Duplicates. Duplicate sample analysis is used to measure analytical precision. This can also be equated to laboratory precision for homogenous samples. Precision criteria are method dependent. If precision criteria are not achieved, corrective action or additional action may be required. Recommended action must be completed before sample data can be reported.

Laboratory Control Duplicate, Spikes & Spiked Duplicates. Spikes and spiked duplicates are used to measure analytical precision and accuracy for the sample matrix selected. Precision and accuracy criteria are method dependent. If precision and accuracy criteria are not achieved, corrective action or additional action may be required. Recommended action must be completed before sample data can be reported.

Serial Dilution (Metals). Serial dilutions of metals samples are analyzed to determine if analytical matrix effects may have impacted the reported data. If the value of the serially diluted samples does not agree with the undiluted value within a method-specified range, the sample matrix may be causing interference, which may lead to either a high or low bias. If the serial dilution criterion is not achieved, it must be flagged to indicate possible bias from matrix effects. SGS Accutest – Orlando *uses this procedure as opposed to post-digestion spike unless contractual obligations absolutely require latter*

Post Digestion Spikes (Metals). Digested samples are spiked and analyzed to determine if matrix interferences are creating biases in the results. It may also be used to determine potential interferences per client's specification. Spike concentration is determined as per analytical method. No action is necessary if the post digestion spike is outside of the method criteria, unless a preparation problem is suspected with the spike, in which case the post digestion spike should remade and reanalyzed.

Surrogate Spikes (Organics). Surrogate spikes are organic compounds that are similar in behavior to the target analytes but unlikely to be found in nature. They are added to all quality control and field samples to measure method performance for each

individual sample. Surrogate accuracy limits are derived from published method specifications or by statistical evaluation of laboratory generated surrogate accuracy data. Accuracy data is compared to the applicable performance limits. If the surrogate accuracy exceeds performance limits, corrective action, as specified in the method or SOP is performed before sample data can be reported.

Internal Standards (Organic Methods). Internal standards are retention time and instrument response markers added to every sample to be used as references for quantitation. Their response is compared to reference standards and used to evaluate instrument sensitivity on a sample specific basis. Internal standard retention time is also compared to reference standards to assure that target analytes are capable of being located by their individual relative retention time.

If internal standard response criteria are not achieved, corrective action or additional action may be required. The recommended action must be completed before sample data can be reported.

If the internal standard retention time criteria are not achieved corrective action or additional action may be required. This may include re-calibration and re-analysis. Additional action must be completed before sample data is reported.

Internal Standards (ICP and ICP-MS Metals). Internal standards are used on ICP instruments to compensate for variations in response caused by differences in sample matrices. This adjustment is performed automatically during sample analysis. The internal standard response of replicated sample analysis is monitored to detect potential analytical problems. If analytical problems are suspected, then the field samples are reanalyzed.

12.4 ***Laboratory Derived Quality Control Criteria.*** Control criteria for in-house methods and client specific modifications that exceed the scope of published methodology are defined and documented prior to the use of the method. The Quality Assurance staff identifies the responsibility for control criteria needs. Control parameters and criteria, based on best technical judgment are established using input provided by the operations staff. These control parameters and criteria are documented and incorporated into the method.

The laboratory derived criteria are evaluated for technical soundness on spiked samples prior to the use of the method on field samples. The technical evaluation is documented and archived by the Quality Assurance staff.

When sufficient data form the laboratory developed control parameter is accumulated, the data is statistically processed and the experimentally derived control limits are incorporated into the method.

12.5 ***Bench Review & Corrective Action***. The bench chemists are responsible for all QC parameters. Before proceeding with sample analysis, they are required to

successfully meet all instrumental QC criteria. They have the authority to perform any necessary corrective action before proceeding with sample analysis. Their authority includes the responsibility for assuring that departures from documented policies and procedures do not occur.

The bench chemists are also responsible for all sample QC parameters. If the sample QC criteria are not achieved, they are authorized and required to perform the method specified corrective action before reporting sample data.

***Data Qualifiers*.** An alpha character coding system is employed for defining use limitations for reported data. These limitations are applied to analytical data by the analyst to clarify the usefulness of the reported data for data user. SGS Accutest - Orlando qualifies data in accordance with program-specific requirements, such as State of Florida DEP, DoD QSM, etc., and these qualifiers are hard-coded in the LIMS on project level. Definitions of common qualifiers could be found at the bottom of the sample report form.

12.6 ***QA Monitoring***. The QA staff prior to client release conducts a spot review of completed data packages. This review includes an examination of QC data for compliance and trends indicative of systematic difficulties. If non-conformances are detected, the QA staff places an immediate stop on the release of the data and initiates corrective action to rectify the situation. The data package is released when the package becomes compliant with all quality requirements.

If the review reveals trends indicative of systematic problems, QA initiates an investigation to determine the cause. If process defects are detected, a corrective action is implemented and monitored for effectiveness.

Performance Limits. The Technical Director is responsible for compilation and maintenance of all precision and accuracy data used for performance limits. Quality control data for all test methods are accumulated and stored in the laboratory information management system (LIMS). Parameter specific QC data is extracted annually and statically processed to eliminate outliers and develop laboratory specific warning limits and confidence limits. The new limits are reviewed and approved by the supervisory staff prior to their use for data assessment. The new limits are used to evaluate QC data for compliance with method requirements for a period of one year. Laboratory generated limits appear on all data reports unless method specifies hard-coded limits (mostly General Chemistry and Metals)

12.7 ***Data Package Review***. SGS Accutest – Orlando employs multiple levels of data review to assure that reported data has satisfied all quality control criteria and that client specifications and requirements have been met. Production departments have developed data review procedures which must be conducted before data is released to the client.

Analytical Review. The analyst conducts the primary review of all data. This review begins with a check of all instrument and method quality control and progresses through sample quality control concluding with a check to assure that the client's requirements have been executed. Analyst checks focuses on a review of qualitative determinations and checks of precision and accuracy data to verify that existing laboratory criteria have been achieved. Checks at this level may include comparisons with project specific criteria if applicable. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter or nonconformance at this stage of review.

Secondary data reviews are performed at the peer level by analysts who have met the qualification criteria for the method in use. Qualification requirements include a valid demonstration of capability and demonstrated understanding of the method SOP. Section supervisors may perform secondary review in-lieu of a peer review Secondary review is performed on 100% of the data produced by their department. It includes a check of all manual calculations; an accuracy check of manually transcribed data from bench sheets to the LIMS, a check of all method and instrument QC criteria, baseline manipulations (if applicable) and a comparison of the data package to client specified requirements. Also included are checks to assure the appropriate methodology was applied and that all anomalous information was properly flagged for communication in the case narrative. Supervisors have the authority to reject data and initiate re-analysis, corrective action, or reprocessing.

All laboratory data requiring manual entry into LIMS system is double-checked by the analysts performing initial data entry and the section supervisor. Verification of supervisory review is indicated on the raw data summary by the supervisor's initials and date.

Electronic data that is manually edited at the bench by the primary analysts is automatically flagged by the instrument data system indicating an override by the analyst. All manual overrides must be verified and approved by a supervisor who initials and dates all manual changes.

Hard copies of manually integrated chromatographic peaks are printed that clearly depict the manually drawn baseline. The hard copy is reviewed and approved by the reviewer (initialed and dated) and included in the data package of all full tier reports or the archived batch records of commercial report packages.

Electronic data that has been committed to the LIMS can only be edited by a manager or supervisor. These edits may be required if needs for corrections are indicated during the final review. An audit record for all electronic changes in the LIMS is automatically appended to the record.

The section leader performs a tertiary review on a spot check basis. This review includes an evaluation of QC data against acceptance criteria and a check of the data

package contents to assure that all analytical requirements and specifications were executed.

Report Generation (Administrative) Review. The report generation group reviews all data and supporting information delivered by the laboratory for completeness and compliance with client specifications. Missing deliverables are identified and obtained from the laboratory. The group also reviews the completed package to verify that the delivered product complies with all client specifications. Non-analytical defects are corrected before the package is sent to the client.

Project Management/Quality Assurance Review. Spot-check data package reviews are performed by the project manager. Project management reviews focus on project specifications. If the project manager identifies defects in the product prior to release, he initiates immediate corrective action to rectify the situation.

The QA Staff reviews approximately 10% of the data produced. The QA review focuses on all elements of the deliverable including the client's specifications and requirements, analytical quality control, sample custody documentation and sample identification. QA reviews at this step in the production process are geared towards systematic process defects, which require procedural changes to effect a corrective action. However, if defects are identified that can be corrected prior to data release, the QA staff returns the package to the laboratory for corrective action. QA data review cannot be used in lieu of a peer level review or a supervisory review.

Data Reporting. Analytical data is released to clients following secondary departmental review. Data release at this stage of the process is limited to electronic information, which is released to clients through a secure, encrypted, password protected, Internet connection.

Hard copy support data is compiled by the report generation group and assembled into the final report. The report is sent to the client following administrative review by report generation staff, and spot-check by QA staff.

All data reports include specified information, which is required to identify the report and its contents. This information includes a title, name and address of the laboratory, a unique report number, total number of pages in the report, clients name and address, analytical method identification, arriving sample condition, sample and analysis dates, test results with units of measurement, authorized signature of data release, statement of applicability, report reproduction restrictions and TNI requirements certification. Subcontracted data is clearly identified.

In the event of report revision date of the revision, nature of revision and identity of the person revising the report must be clearly stated in the body of the report. All levels of deliverables incorporate letter(s) of report reissue into all subsequent reports. This letter(s) is addressed to the client and briefly outlines reasons for report revision.

12.8 ***Electronic Data Reduction.*** Raw data from sample analysis is entered into the laboratory information management system (LIMS) using automated processes or manual entry. Final data processing is performed by the LIMS using procedures developed by the Company.

All LIMS programs and internally developed software (including Excel spreadsheets) are tested and validated prior to use to assure that they consistently produce correct results. Validation testing is performed by the Information Technology Staff. The testing procedures are documented in an SOP. Programs are not approved for use until they have demonstrated that they are capable of performing the required calculations.

12.9 ***Representativeness***. Data representativeness is based on the premise that qualitative and quantitative information developed for field samples is characteristic of the sample that was collected by the client and analyzed in the laboratory. The laboratory objective for representativeness defines data as representative if the criteria for all quality parameters associated with the analysis of the sample are achieved.

12.10 ***Comparability***. Analytical data is defined as comparable when data from a sample set analyzed by the laboratory is representatively equivalent to other sample sets analyzed separately regardless of the analytical logistics. The laboratory will achieve 100% comparability for all sample data which meets the criteria for the quality parameters associated with its analysis using the method requested by the client.

13.0 CORRECTIVE ACTION SYSTEM

Requirement. The laboratory must have polices and procedures for correcting defective processes, systematic errors, and quality defects, which enables the staff to systematically improve product quality. The system must include procedures for communicating items requiring corrective action, corrective action tracking procedures, corrective action documentation, monitoring of effectiveness, and reports to management. The system must be documented in a standard operating procedure.

13.1 ***Procedure***. Corrective action is the step that follows the identification of a process defect. The type of defect determines the level of documentation, communication, and training necessary to prevent re-occurrence of the defect or non-conformance.

Routine Corrective Action. Routine corrective action is defined as the procedures used to return out of control analytical systems back to control. This level of corrective action applies to all analytical quality control parameters or analytical system specifications.

Bench analysts have full responsibility and authority for performing routine corrective action. The resolution of defects at this level does not require a procedural change or staff re-training. The analyst is free to continue work once corrective action is complete and the analytical system has been returned to control. Documentation of routine corrective action is limited to bench logbook or maintenance logbook comment.

Process Changes. Corrective actions in this category require procedural modifications. They may be the result of systematic defects identified during audits, the investigation of client inquiries, failed proficiency tests, product defects identified during data review, or method updates. Resolution of defects of this magnitude requires formal identification of the defect, development and documentation of a corrective action plan, and staff training to communicate the procedural change.

Technical Corrective Action. Technical corrective action encompasses routine corrective action performed by bench analysts for out of control systems and corrective actions performed for data produced using out of control systems. Technical corrective action for routine situations is conducted using the procedures detailed above.

Non-routine corrective actions apply to situations where the bench analysts failed to perform routine corrective action before continuing analysis. Supervisors and Department Managers perform corrective action in these situations. Documentation of all non-routine corrective actions is performed using the corrective action system.

Sample re-analysis is conducted if sufficient sample and holding time remain to repeat the analysis using an in-control system. If insufficient sample or holding time remains, the data is processed and qualifiers applied that describe the out of control situation. The occurrence is further documented in the case narrative and in the corrective

action response. The corrective action must include provisions for retraining the analysts who failed to perform routine corrective action.

13.2 ***Documentation & Communication***. Routine corrective actions are documented as part of the analytical record. Notations are made in the comments section of the analytical chronicle or data sheet detailing the nonconformance. Continuation of the analysis indicates that return to control was successful.

Corrective actions for process changes are documented, tracked and monitored for effectiveness. Corrective actions may be initiated by any supervisor or senior staff member by completing the corrective action form in Corrective Action database

The corrective action database is an Access application. The initiator generates the corrective action investigation form, which is documented, tracked, distributed to responsible parties and archived through the application. The application assigns a tracking number initiation data and due date to each corrective action initiated and copies the corrective action form to the corrective action database. The application also distributes an E-mail message containing the form to the responsible parties for resolution.

Corrective Action system employs Deficiency – Root Cause – Immediate Fix – Corrective action approach, further divided into categories of Analytical Error, Omission Error, Random Error, Systemic Error and Training Issue.

The responsible party develops and implements the procedural change. Existing documentation such as SOPs are edited to reflect the change. The affected staff is informed of the procedural change through a formal training session. The training is documented and copies are placed into individual training files. The corrective action form is completed and closed in CA database.

Initial and completed corrective action forms are maintained in the Corrective Action directory. This information is archived daily. Copies of training records describing corrective actions are appended to the involved individuals training files.

Monitoring. The QA Staff monitors the implemented corrective action until it is evident that the corrective action has been effective and the systematic deficiency has been eliminated. The corrective action database is updated by QA to reflect closure of the corrective action. The QA staff also assigns an error code to the corrective action for classification of the type of errors being committed.

If QA determines that the corrective action procedure has not effectively remedied the deficiency, the process continues with a re-initiation of the corrective action. Corrective action continues until the defective process is eliminated. If another procedural change is required, it is treated as a new corrective action, which is documented and monitored using established procedures.

Client Notification. Defective processes, systematic errors, and quality defects, detected during routine audits may have negative impacts on data quality. In some cases, data that has been released to clients may be affected. If defective data has been released for use, SGS Accutest – Orlando will notify the affected clients of the defect and provide specific details regarding the magnitude of the impact to their data.

14.0 PROCEDURES FOR EXECUTING CLIENT SPECIFICATIONS

Requirement. Systems must be established for evaluating and processing client specifications for routine and non-routine analytical services. The systems must enable the client services staff to identify, evaluate, and document the requested specifications to determine if adequate resources are available to perform the analysis. The system must include procedures for communicating the specifications to the laboratory staff for execution and procedures for verifying the specifications have been executed.

14.1 ***Client Specific Requirements***. The project manager is the primary contact for clients requesting laboratory services. Client specifications are communicated using several mechanisms. The primary source of information is the client's quality assurance project plan (QAPP) which details analytical and quality control specifications for the project. In the absence of a QAPP, projects specifications can also be communicated using contracts, letters of authorization, or letters of agreement, which may be limited to a brief discussion of the analytical requirements and the terms and conditions for the work. These documents may also include pricing information, liabilities, scope of work, in addition to the analytical requirements. QAPPs include detailed analytical requirements and data quality objectives, which supersede those found in the referenced methods. This information is essential to successful project completion.

Laboratory also reviews its Accreditation status to evaluate whether it is possible to accept proposed project. Discrepancies must be resolved before the work commences.

The client services staff provides additional assistance to clients who are unsure of the specifications they need to execute the sampling and analysis requirements of their project. They provide additional support to clients who require assistance in results interpretation as needed, provided they possess the expertise required to render an opinion.

The project manager is responsible for obtaining project documents, which specify the analytical requirements. Following project management review, copies are distributed to the QA staff and the appropriate departmental managers for review and comment. The original QAPP is numbered with a document control number and filed in a secure location.

14.2 ***Requirements for Non-Standard Analytical Specifications***. Client requirements that specify departures from documented policies, procedures, or standard specifications must be submitted to SGS Accutest – Orlando in writing. These requirements are reviewed and approved by the technical staff before the project is accepted. Once accepted, the non-standard requirements become analytical specifications, which follow the routine procedure for communicating client specifications. Departures from documented policies, procedures, or standard specifications that do not follow this procedure are not permitted.

Exception Policy: With respect to the quality system, incoming non-conforming product refers to received samples that do not meet requirements of custody documentation, are improperly packaged or stored or are contaminated. An internal non-conformance refers to a problem, caused internally due to improper handling of samples, improper sampling methods, and equipment malfunction or data management errors. The individual who identifies the incoming non-conformance is responsible for notifying the project manager. The project manager resolves the issue with the client. The individual who recognizes an internal non-conformance is responsible for initiating corrective action

Departures from standard practices, policies and specifications are reviewed and approved by Technical Director, QA Officer and by Project Manager of the project affected.

Corrective & Preventative Action: Once a quality problem has been identified, the analytical or review process stops, until the reason is identified. Primary responsibility for identifying the cause of the problem rests with the instrument operator. Other staff may be called on to assist in reaching the root cause. The problem prevention tracking system, using Corrective Action Tracking Records, provides a method to track systemic problems until resolved/removed. The QA Officer is responsible for the record management with respect to the disposition of problems.

Deviations that do not limit themselves to a single department and/or client are cited on Corrective Action Record. This may include but not limited to: sample arrival outside of EPA specified holding time, analysis completion outside of EPA specified holding time (with explanation of the reason), inconsistencies between chain of custody and cooler contents, including labeling errors, improper preservation, etc.

Deviations from analytical methods' SOP's are reported by the Analyst to the Section Leader. Single occurrences warrant completion of Corrective Action Tracking record, repetitive occurrences may indicate that either an additional training session is in order, or that the SOP does not reflect proper laboratory practice. Training session is conducted by the Technical Director or by QA Officer. In case where SOP does not reflect current laboratory practice, SOP review and correction process may be initiated.

14.3 ***Evaluation of Resources.*** A resource evaluation is completed prior to accepting projects submitted by clients. The evaluation is initiated by the client services staff receives project requirements (usually in the form of QAPjP) and distributes these requirements to the laboratory departments affected. The specifications are evaluated by the department managers from a scheduling and hardware resources perspective. The project is not accepted unless the department managers have the necessary resources to execute the project according to client specifications.

14.4 ***Documentation***. New projects are initiated using a project set up form, which is completed prior to the start of the project. This form details all of the information needed to correctly enter the specifications for each client sample into the laboratory information management system (LIMS, see example). The form includes data reporting requirements, billing information, data turnaround times, QA level, state of origin, and comments for detailing project specific requirements. The project manager is responsible for obtaining this information from the client and completing the form prior to sample arrival and login.

Sample receipt triggers project creation and the login process. The information on the set-up form is entered into the LIMS immediately prior to logging in the first sample. The set up form may be accompanied by a quotation, which details the analytical product codes and sample matrices. These details are also entered into the LIMS during login.

Special information is distributed to the laboratory supervisors and login department in electronic or hardcopy format upon project setup. All project specific information is retained by the project manager in a secure file. The project manager maintains a personal telephone log, which details conversations with the client regarding the project.

14.5 ***Communication***. A pre-project meeting is held between client services and the operations managers to discuss the specifications described in the QAPjP and/or related documents. Project logistics are discussed and finalized and procedures are developed to assure proper execution of the client's analytical specifications and requirements. Questions, raised in the review meeting, are discussed with the client for resolution. Exceptions to any requirements, if accepted by the client, are documented and incorporated into the QAPjP or project documentation records.

Non-standard specifications for individual clients are documented in the LIMS at the client account level. Once entered into the LIMS, these specifications become memorialized for all projects related to the client account. Upon sample arrival, these specifications are accessed through a terminal or printed as a hard copy and stored in a binder for individuals who require access to the specification. Specifications that are not entered into the LIMS are prohibited unless documented in an interdepartmental memo, which clearly identifies the project, client and effective duration of the specification.

14.6 ***Operational Execution***. A work schedule is prepared for each analytical department on a daily basis. Analytical specifications from recently arrived samples have now been entered into the LIMS database. The database is sorted by analytical due date and holding time, into product specific groups. Samples are scheduled for analysis by due date and holding time. The completed schedule, which is now defined as a work list, is printed. The list contains the client requested product codes and specifications required for the selected sample(s). Special requirements are communicated to the analyst using the comments section or relayed through verbal instructions provided by

the supervisor. The bench analyst assumes full responsibility for performing the analysis according to the specifications printed on the work sheet.

14.7 ***Verification***. Prior to the release of data to the client, laboratory section managers and the report generation staff review the report and compare the completed product to the client specifications documentation to assure that all requirements have been met. Project managers perform a spot check of projects with unique requirements to assure that the work was executed according to specifications.

15.0 CLIENT COMPLAINT RESOLUTION PROCEDURE

Requirement. A system for managing and reconciling client complaints must be implemented in the laboratory. The system must include procedures for documenting client complaints and communicating the complaint to the appropriate department for resolution. The system must also include a quality assurance evaluation to determine if the complaint is related to systematic defects requiring process changes.

15.1 ***Procedure***. Client complaints are communicated to client services representatives, quality assurance staff, or senior management staff for resolution. The individual receiving the complaint retains the responsibility for documentation and communicating the nature of the complaint to the responsible department(s) for resolution. The responsible party addresses the complaint. The resolution is communicated to quality assurance (QA) and the originator for communication to the client. QA reviews the complaint and resolution to determine if systematic defects exist. If systematic defects are present, QA works with the responsible party to develop a corrective action that eliminates the defect.

Documentation. Client's complaints are documented by the client service representative receiving the complaint. A record of the telephone conversation is maintained by client services. Client service staff enters the complaint into Data Challenge database or Client Complaint database, depending on the nature of complaint. These databases are cross-linked with corrective action database – see sec. 13. Complaint is communicated to the production departments concerned via auto e-mail. The complaint resolution is documented in the database by the responsible party and resultant e-mail returned to the originator. QA staff is copied on the correspondence.

15.2 ***Corrective Action***. Responses to Data Challenges/Client Complaints are required from the responsible party. At a minimum, the response addresses the query and provides an explanation to the complaint. Corrective action may focus on the single issue expressed in the complaint. Corrective action may include job case narrative generation, reprocessing of data, editing of the initial report, and re-issue to the client. If the QA review indicates a systematic error, process modification is required. The defective process at the root of the complaint is changed. SOPs are either created or modified to reflect the change. The party responsible for the process implements process changes.

15.3 ***QA Monitoring***. Process changes, implemented to resolve systematic defects, are monitored for effectiveness by QA. If monitoring indicates that the process change has not resolved the defect, QA works with the department management to develop and implement an effective process. If monitoring indicates that the defect has been resolved, monitoring is slowly discontinued. Continued monitoring is incorporated as an element of the annual system audit and annual Management Report (see 18.8).

16.0 CONTROL OF NONCONFORMING PRODUCT

Requirement: Policies and procedures have been developed and implemented that describe the procedures employed by the laboratory when any aspect of sample analysis or data reporting do not conform to established procedures or client specifications. These procedures include steps to ensure that process defects are corrected and affected work is evaluated to assess its impact to the client.

Procedure. Nonconforming product is identified through multiple channels, such as second level analytical data review, routine internal review and audit practices, external auditing or through client inquiry. Responsibility and authority for the management of the non-conforming product is directly defined by a nature of a non-conformance. For example, non-conformances resulting from internal and external reviews are evaluated and managed by QA Staff. Corrective Action items are issued and followed to completion and verification that defect is prevented from reoccurring. Non-conformances stemming from client inquiry are managed by Project Management staff with QA staff oversight.

Data associated with out-of compliance QC are evaluated by bench personnel and section supervisors. The analyst has the authority and responsibility to perform corrective action for any out-of-control parameter or nonconformance at this stage of review.

If non-conformances are detected, the QA staff places an immediate stop on the release of the data and initiates corrective action to rectify the situation

Non-conformances and their significance are communicated in case narrative and sample report footnotes. Case narrative comments and sample repot footnotes must state the impact on data quality.

Corrective Action. The outcome of the evaluation dictates the course of action. The type of defect determines the level of documentation, communication, and training necessary to prevent re-occurrence of the defect or non-conformance This may include at a minimum client notification, but may also include corrective action. Immediate corrective action is performed using the SOP-specified procedures. However, additional action may be required including cessation of analysis and withholding and/or recalling data reports. If the evaluation indicates that nonconforming data may have been issued to clients, the client is immediately notified and data may be recalled following the procedures specified in respective SOPs. If work has been stopped because of a nonconformance, the Laboratory Director is the only individual authorized to direct a resumption of analysis.

Non-conformances caused by systematic process defects require retraining of the personnel involved as an element of the corrective action solution. Routine corrective actions are documented as part of the analytical record.

17.0 CONFIDENTIALITY PROTECTION PROCEDURES

Requirements: Policies and procedures are required to protect client data from release to unauthorized parties or accidental release of database information through accidental electronic transmission or illegal intrusion. These policies must be communicated to clients and staff. Electronic systems must be regularly evaluated for effectiveness.

17.1 ***Client Anonymity***. Information related to the Company's clients is granted to employees on a "need to know" basis. An individual's position within the organization defines his "need to know". Individuals with "need to know" status are given password access to systems that contain client identity information and access to documents and document storage areas containing client reports and information. Access to client information by individuals outside of the Company is limited to the client and individuals authorized by the client.

Individuals outside of the Company may obtain client information through subpoena issued by a court of valid jurisdiction. Clients are informed when subpoenas are received ordering the release of their information.

17.2 ***Documents***. Access to client documents is restricted to employees in need to know positions. Copies of all client reports are stored in secure archive with restricted access. Reports and report copies are distributed to individuals who have been authorized by the client to receive them. Documents are not released to third parties without verbally expressed or written permission from the client.

17.3 **Confidential Business Information (CBI).** Operational documents including SOPs, Quality Manuals, personnel information, internal operations statistics, and laboratory audit reports are considered confidential business information. Strict controls are placed on the release of this information to outside parties.

Release of CBI to outside parties or organizations may be authorized upon execution of a confidentiality agreement between SGS Accutest – Orlando and the receiving organization or individual. CBI information release is authorized for third party auditors and commercial clients in electronic mode as Adobe Acrobat .PDF format only. See also Sec. 6.5.

17.4 ***Electronic Data***.

Database Intrusion. Direct database entry is authorized for employees of SGS Accutest – Orlando only on a need to know basis. Entry to the database is restricted through a user specific multiple password entry system. Direct access to the database outside of the facility is possible through a VPN connection. A unique user and password is required for access to the local area network. A second unique password is required to gain access to the database. All Passwords are required to be changed

semiannually. The staff receives read or write level authorization on a hierarchical privilege basis.

Internet Access. Access to client information is through an HTTPS Web application only. It does not contain a mechanism that allows direct access to the database. Clients can gain access to their data only using a series of SGS Accutest assigned accounts, and client specific passwords. The viewable data, which is encrypted during transmission, consists of an extraction of database information only.

Client Accessibility. Accessibility to client data delivered via electronic means follows strict protocols to insure confidentiality. Clients accessing electronic data are assigned a company account. The account profile, which is established by the MIS staff, grants explicit access to explicit information pertaining to the client's project activity. Passwords are assigned on an individual basis within a client account. These accounts can be activated or deactivated by the MIS staff only.

17.5 ***Information Requests***. Client specific data or information is not released to third parties without verbally expressed or written permission from the client. Written permission is required from third parties, who contact the Company directly for the release of information. Verbal requests will be honored only if they are received directly from the client. These requests must be documented in a record of communication maintained by authorized recipient.

17.6 ***Transfer of Records***. Archived data, which has previously been reported and transmitted to clients, is the exclusive property of SGS Accutest. In the event of a cessation of business activities due to business failure or sale, The Company's legal staff will be directed to arrange for the final disposition of archived data.

The final disposition of archived data will be accomplished using the approach detailed in the following sequence:

1. All data will be transferred to the new owners for the duration of the required archive period as a condition of sale.

2. If the new owners will not accept the data or the business has failed, letters will be sent to clients listed on the most recent active account roster offering them the option to obtain specific reports (identified by SGS Accutest – Orlando Job Number) at their own expense.

3. A letter will be sent to the TNI accrediting authority with organizational jurisdiction over the company offering them the option to obtain all unclaimed reports at their own expense.

4. All remaining archived data will be recycled using the most expedient means possible.

18.0 QUALITY AUDITS AND SYSTEM REVIEWS

Requirement: The quality assurance group will conduct regularly scheduled audits of the laboratory to assess compliance with quality system requirements, technical requirements of applied methodology, and adherence to documentation procedures. The information gathered during these audits will be used to provide feedback to senior management and perform corrective action where needed for quality improvement purposes.

18.1 ***Quality Systems Review***. Quality system audits are performed annually by the Quality Assurance Director for the Company President. In this audit, the laboratory is evaluated for compliance with the Laboratory Quality Systems Manual (LQSM) and the quality system standards of TNI. Findings, which indicate non-compliance or deviation from the LQSM, are flagged for corrective action. Corrective actions require either a return to compliance or a plan change to reflect an improved quality process. The QA Officer is responsible for making and documenting changes to the LQSM. These changes are reviewed by the Laboratory Director and Technical Director prior to the approval of the revised system.

18.2 ***Quality System Audits.*** Quality system audits are conducted to evaluate the effectiveness and laboratory compliance with individual quality system elements. These audits are conducted on an established schedule. Audit findings are documented and communicated to the management staff and entered into the corrective action system for resolution. If necessary, retraining is conducted to assure complete understanding of the system requirements.

18.3 ***Technical Compliance Audits***. Technical compliance audits are performed throughout the year following the established schedule. Selected analytical procedures are evaluated for compliance with standard operating procedures (SOPs) and method requirements. If non-conformances exist, the published method serves as the standard for compliance. SOPs are edited for compliance if the document does not reflect method requirements. Analysts are trained to the new requirements and the process is monitored by quality assurance. Analysts are retrained in method procedures if an evaluation of bench practices indicates non-compliance with SOP requirements.

18.4 ***Documentation Audits***. Documentation audits are conducted periodically. This audit includes a check of measurement processes that require manual documentation and non-analytical logbook review. It also includes checks of data archiving systems and a search to find and remove any inactive versions of SOPs that may still be present in the laboratory and being accessed by the analysts. Non-conformances are corrected on the spot. Procedural modifications are implemented if the evaluation indicates a systematic defect.

18.5 ***Corrective Action Monitoring***. Defects or non-conformances that are identified during client or internal audits are shared with management and entered into CA database for attention by the responsible party. Audit findings are corrected through

process modifications and/or retraining. Once a corrective action has been designed and implemented, it is monitored for compliance on a regular basis by the QA staff. Monitoring of the corrective action continues until satisfactory implementation has been verified.

18.6 ***Preventive Action.*** Laboratory systems or processes, which may be faulty and pose the potential for nonconformances, errors, confusing reports or difficulties establishing traceability may be identified during internal audits. These items are highlighted for systematic change using the corrective action system and managed to resolution using appropriate procedures for corrective action.

18.7 ***Client Notification.*** Defective processes, systematic errors, and quality defects detected during routine audits may have negative impact on data quality. In some cases, data that has been released to the client may be affected. If defective data has been released for use, SGS Accutest – Orlando will immediately notify the affected clients of the defect and provide specific details regarding the magnitude of the impact to their data.

18.8 ***Management Reports***. Formal reports of all audit activities are prepared for the management staff. These reports are prepared annually. The report details the status of the Quality System.

The formal report also addresses the following topics:

- *the suitability of policies and procedures;*
- *reports from managerial and supervisory personnel;*
- *the outcome of recent internal audits;*
- *corrective and preventive actions;*
- *assessments by external bodies;*
- *the results of inter-laboratory comparisons or proficiency tests;*
- *changes in the volume and type of the work;*
- *customer feedback;*
- *complaints;*
- *recommendations for improvement;*
- *other relevant factors, such as quality control activities, resources, and staff training.*

19.0 HEALTH AND SAFETY

Requirement. The company operates a formal health and safety program that complies with the requirements of the Occupational Health and Safety Administration. The program consists of key policies and practices that are essential to safe laboratory operation. All employees are required to receive training on the program elements. Job specific training is conducted to assure safe practices for specific tasks. All employees are required to participate in the program, receive initial and annual training, and comply with the program requirements. All plan and program requirements are detailed in the Health and Safety Program Manual.

19.1 Policy. SGS Accutest will provide a safe and healthy working environment for its employees and clients while protecting the public and preserving the Company's assets and property. The company will comply with all applicable government regulations pertaining to safety and health in the laboratory and the workplace.

The objective of the SGS Accutest Health and Safety Program is to promote safe work practices that minimize the occurrence of injuries and illness to the staff through proper health and safety training, correct laboratory technique application and the use of engineering controls.

19.2 Responsibilities. The Health and Safety Program assists managers, supervisors and non-supervisory employees in control of hazards and risks to minimize the potential for employee and client injuries, damage to client's property and damage or destruction to SGS Accutest's facilities.

The Health and Safety Officer is responsible for implementing the Program's elements and updating its contents as necessary. He also conducts periodic audits to monitor compliance and assess the program's effectiveness and is also responsible for creating and administering safety training for all new and existing employees.

The employee is responsible for following all safety rules established for their protection, the protection of others and the proper use of protective devices provided by the Company. The employee is also expected to comply with the requirements of the program at all times. Department Managers and Supervisors are responsible for ensuring the requirements of the Safety Program are practiced daily. The Company President retains the ultimate responsibility for the program design and implementation.

19.3 Program Elements. The SGS Accutest Health and Safety Program consists of key program elements that compliment the company's health and safety objective. These elements form the essence of the health and safety policy and assure that the objectives of the program are achieved.

Safety Education and Training and Communication. Training is conducted to increase the staff's awareness of laboratory hazards and their knowledge of the safety

practices and procedures required to protect them from those hazards. It is also used to communicate general safety procedures required for safe operation in a chemical laboratory.

Initial health and safety training for new employees is conducted during new employee orientation and administered through SGS Accutest University database.. The training focuses on the SGS Accutest Safety and Health Program and includes specific training for the hazards that may be associated with the employees' duties. Training is also conducted for all program elements focusing on general, acceptable, laboratory safety procedures. Targeted training is conducted to address hazards or safety procedures that are specific to individual employee's work assignments. All training activities are documented and archived in individual training folders. A health and safety training inventory is maintained in the training database.

SGS Accutest - Orlando maintains personnel trained in HAZWOPER, DOT and HazMat operations, as well as respirator fit certification.

Safety Officer. The safety officer provides the employees with an opportunity to express their views and concerns on safety issues in an environment where those concerns will be addressed to ensure that the interests of the company and the well being of the employee are protected. Safety Officer is entrusted with elevating the level of safety awareness among their peers.

Hazard Identification and Communication. The hazard communication program enables employees to readily identify laboratory hazards and the procedures to protect themselves from those hazards. This program complies with OSHA's Hazard Communication Standard, Title 29 Code of Federal Regulations 1910.1200 that requires the company to adopt and adhere to the following key elements:

- Material Safety Data Sheets (MSDS) and/or Safety Data Sheets (SDS) must be available to any employee wishing to view them,
- The Company must maintain a Hazardous Chemicals Inventory (by location), which is updated on an annual basis,
- Containers are properly labeled,
- All employees must be provided with annual Personal Protection, Hazard Communication and Right to Know training,

Chemical Hygiene Plan. The Chemical Hygiene Plan complies with the requirements of the Occupational Safety and Health Administration's Occupational Exposure to Hazardous Chemicals in the Laboratory Standard, 29 CFR 1910.1450. This plan establishes procedures, identifies safety equipment, personal protective equipment, and work practices that protect employees from the potential health hazards presented by hazardous chemicals in the laboratory if properly used and/or applied.

Emergency Action & Evacuation Plan. The Emergency Action and Evacuation Plan details the procedures used to protect and safeguard SGS Accutest – Orlando employees and property during emergencies. Emergencies are defined as fires or explosions, gas leaks, building collapse, hazardous material spills, emergencies that immediately threaten life and health, bomb threats and natural disasters such as floods, hurricanes or tornadoes. The plan identifies and assigns responsibility for executing specific roles in situations requiring emergency action.

Lockout/Tagout Plan. Lockout/tagout procedures have been established to assure that laboratory employees and outside contractors take steps to render equipment inoperable and/or safe before conducting maintenance activities. The plan details the procedures for conducting maintenance on equipment that has the potential to unexpectedly energize, start up, or release energy or can be operated unexpectedly or accidentally resulting in serious injury to employees. The plan ensures that employees performing maintenance render the equipment safe through lock out or tag out procedures.

Personal Protection Policy. Policies have been implemented which detail the personal protection requirements for employees. The policy includes specifications regarding engineering controls, personal protective equipment (PPE), hazardous waste, chemical exposures, working with chemicals and safe work practices. Safety requirements specific to processes or equipment are reviewed with the department supervisor or the Health and Safety Officer before beginning operations.

Emergency Preparedness Plan. This plan identifies the actions to be taken by SGS Accutest – Orlando staff in the event of terrorism or terrorist actions, to ensure the safety of the employees and the facility. The plan describes the building security actions coinciding with the “Alert Condition”, designated by the Department of Homeland Security.

Appendix I

Glossary of Terms

GLOSSARY OF TERMS

Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents.

Accreditation: the process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of TNI program, this process is a voluntary one.

Accuracy: the degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator.

Analyst: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality.

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis.

Audit: a systematic evaluation to determine the conformance to quantitative *and qualitative* specifications of some operational function or activity.

Batch: environmental samples that are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A **preparation batch** is composed of one to 20 environmental samples of the same quality-system matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples.

Blank: a sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results.

Blind Sample: a sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

Case Narrative: a statement of non-conformances associated with particular data report

Calibration: to determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements.

Calibration Curve: the mathematical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response.

Calibration Method: a defined technical procedure for performing a calibration.

Calibration Standard: a substance or reference material used to calibrate an instrument.

Certified Reference Material (CRM): a reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body.

Chain of Custody: an unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples.

Clean Air Act: the enabling legislation in 42 U.S.C. 7401 *et seq.*, Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and to enforce them.

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): the enabling legislation in 42 U.S.C. 9601-9675 *et seq.*, as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601*et seq.*, to eliminate the health and environmental threats posed by hazardous waste sites.

Confirmation: verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to second column confirmation, alternate wavelength, derivatization, mass spectral interpretation, alternative detectors or, additional cleanup procedures.

Conformance: an affirmative indication or judgement that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements.

Corrective Action: the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence.

Data Audit: a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form.

Demonstration of Capability: a procedure to establish the ability of the analyst to generate acceptable accuracy.

Document Control: the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed.

Duplicate Analyses: the analyses or measurements of the variable of interest performed identically on two sub-samples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory.

Federal Water Pollution Control Act (Clean Water Act, CWA): the enabling legislation under 33 U.S.C. 1251 *et seq.*, Public Law 92-50086 Stat. 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance.

Field of Testing: TNI's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required submit to only that portion of the accreditation process not previously addressed (see TNI, section 1.9ff).

Holding Times (Maximum Allowable Holding Times) the maximum times that samples may be held prior to analysis and still be considered valid or not compromised.

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample): a sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes from a source independent of the calibration standards or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

Matrix (or Quality System Matrix): the component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other leachates.

Drinking Water: any aqueous sample that has been designated a potable or potential potable water source. Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt-water source such as the Great Salt Lake. Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Biological Tissue, Biota: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter, or other device.

Matrix Spike (spiked sample or fortified sample): a sample prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix Spike Duplicate (spiked sample or fortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Method Blank: a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest, which is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses.

Method Detection Limit: the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

National Institute of Standards and Technology (NIST): an agency of the US Department of Commerce's Technology Administration that is working with EPA, States, TNI, and other public and commercial entities to establish a system under which private sector companies and interested States can be accredited by NIST to provide NIST-traceable proficiency testing (PT) to those laboratories testing drinking water and wastewater.

The NELAC institute (TNI): a voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories.

TNI Standards: the plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the The NELAC Institute.

Performance Audit: the routine comparison of independently obtained *qualitative and quantitative* measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory.

Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms.

Preservation: refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample.

PT Fields of Testing: TNI's approach to offering proficiency testing by regulatory or environmental program, matrix type, and analyte.

Proficiency Testing: a means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source.

Proficiency Test Sample (PT): a sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria.

Quality Assurance: an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence.

Quality Control: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users.

Quality Manual: a document stating the management policies, objectives, principles, oganizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users.

Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC.

Quantitation Limits: the maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user.

Range: the difference between the minimum and the maximum of a set of values.

Raw Data: any original factual information from a measurement activity or study recorded in a laboratory notebook, worksheets, records, memoranda, notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography, microfilm or microfiche copies, computer printouts, magnetic media, including dictated observations, and recorded data from automated instruments. If exact copies of raw data have been prepared (e.g., tapes which have been transcribed verbatim, data and verified accurate by signature), the exact copy or exact transcript may be submitted.

Reagent Blank (method reagent blank or method blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps.

Reference Material: a material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials.

Reference Method: a method of known and documented accuracy and precision issued by an organization recognized as competent to do so.

Reference Standard: a standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived.

Replicate Analyses: the measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval.

Requirement: denotes a mandatory specification; often designated by the term “shall”.

Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq.* (1976), that gives EPA the authority to control hazardous waste from the “Cradle-to-grave”, including its generation, transportation, treatment, storage, and disposal.

Safe Drinking Water Act (SDWA): the enabling legislation, 42 USC 300f *et seq.* (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations.

Sample Duplicate: two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis.

Spike: a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

Standard: the document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of TNI and meets the approval requirements of TNI procedures and policies.

Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 *et seq.*, (1976), that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture.

Traceability: the property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons.

United States Environmental Protection Agency (EPA): federal governmental agency with the responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends.

Validation: the process of substantiating specified performance criteria.

Verification: confirmation by examination and provision of evidence that specified requirements have been met.

NOTE: In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment. The result of verification leads to a decision either to restore in service, to perform adjustment, to repair, to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Appendix II

Analytical Capabilities

TNI-Accredited Fields of Testing

Method Type	Method Number	Regulatory Program
Organics		
EDB and DBCP	EPA 504.1	Drinking Water
Perfluorinated Carboxylic Acids and Sulfonates	EPA 537	Drinking Water
Metals		
ICP: General	EPA 200.7, 1994	Drinking Water
Cold Vapor Mercury	EPA 245.1, 1994	Drinking Water
Organics		
EDB and DBCP	EPA 504, SW846 8011**	Non-Potable Water
Volatile Organics	EPA 624**, SW846 8260B, C**	Non-Potable Water
Semi-Volatile Organics	EPA 625**, SW846 8270D**	Non-Potable Water
Semi-Volatile Organics	SW846 8270D SIM**	Non-Potable Water
Purgeable Aromatics	EPA 602**, SW846 8021A**	Non-Potable Water
Chlorinated Pesticides & PCBs	EPA 608**, SW846 8081B**, 8082A**	Non-Potable Water
Poly-Aromatic Hydrocarbons	EPA 610**, SW846 8310**	Non-Potable Water
Nitroaromatics	SW846 8091**	Non-Potable Water
Explosives	SW846 8330A**, 8332**	Non-Potable Water
Explosives	SW846 8330B**,	Non-Potable Water
Chlorinated Herbicides	SW846 8151A**	Non-Potable Water
Organophosphorus Pesticides	SW846 8141B**	Non-Potable Water
Perchlorate	SW-846 6850	Non-Potable Water
Perfluorinated Carboxylic Acids and Sulfonates	EPA 537 MOD** (ALS MS014)	Non-Potable Water
Dissolved Gases	RSK SOP 147-175**	Non-Potable Water
Alcohols	SW846 8015C,D**	Non-Potable Water
Gasoline Range Organics	SW846 8015C,D**	Non-Potable Water
Diesel Range Organics	SW846 8015C,D**	Non-Potable Water
Total Petroleum Hydrocarbons	FLPRO**	Non-Potable Water
Tennessee EPH	TN-EPH**	Non-Potable Water
Tennessee GRO	TN-GRO**	Non-Potable Water
Wisconsin DRO	WI-DRO**	Non-Potable Water
Petroleum Hydrocarbons	Iowa OA-1**	Non-Potable Water
Petroleum Hydrocarbons	Iowa OA-2**	Non-Potable Water
Volatile Petro. Hydrocarbons	Massachusetts VPH, 2004**	Non-Potable Water

Method Type	Method Number	Regulatory Program
Extractable Petro. Hydrocarbons	Massachusetts EPH, 1998**	Non-Potable Water
Total Petroleum Hydrocarbons	TX-1005**	Non-Potable Water
Acrylamide	SW846 8316	Non-Potable Water
Metals		
ICP: General – EPA WW	EPA 200.7**, 1994; SW-846 6010C**6010D**	Non-Potable Water
ICP/MS: General – EPA WW	EPA 200.8**, 1994; SW-846 6020A**	Non-Potable Water
Cold Vapor Mercury – EPA WW	EPA 245.1, 1994; SW-846 7470A**	Non-Potable Water
Inorganic WetChem		
Alkalinity	SM2320B-11**	Non-Potable Water
CBOD	SM5210B-11	Non-Potable Water
COD	SM5220C-11	Non-Potable Water
BOD	SM5210B-11	Non-Potable Water
Color, Apparent	SM2120B-11	Non-Potable Water
Ion Chromatography (Bromide, Fluoride, Chloride, Sulfate, Nitrite, Nitrate,) – Aqueous	EPA 300.0**, SW846 9056A**	Non-Potable Water
Nitrate/Nitrite	EPA 353.2**	Non-Potable Water
Total Kjeldahl Nitrogen	EPA 351.2**	Non-Potable Water
Ammonia	EPA 350.1**	Non-Potable Water
Oil & Grease, Gravimetric – AQ	EPA 1664A**, SW846 9070A**	Non-Potable Water
Orthophosphate	EPA 365.3**	Non-Potable Water
pH by electrode (Waters)	SM4500H+B-11**; SW846 9040C**	Non-Potable Water
Specific Conductance	EPA 120.1**	Non-Potable Water
Sulfide	SM4500S=F-11**	Non-Potable Water
Total Dissolved Solids	SM2540C-11**	Non-Potable Water
Total Organic Carbon	SM5310B-11, SW846 9060A**	Non-Potable Water
Total Phosphorus	EPA 365.3**	Non-Potable Water
Total Solids	SM2540B-11**	Non-Potable Water
Total Suspended Solids	SM2540D-11**	Non-Potable Water
Turbidity	EPA 180.1	Non-Potable Water
Total CN	EPA 335.4, SW846 9012B**	Non-Potable Water
Un-Ionized Ammonia - calculation	FDEP SOP10/03/83	Non-Potable Water

Method Type	Method Number	Regulatory Program
Calcium Hardness by Calculation	SM2340B-11	Non-Potable Water
Hardness, Total by Calculation	SM2340B-11	Non-Potable Water
MBAS (Anionic Surfactants as)	SM5540C-11	Non-Potable Water
Corrosivity & pH – aqueous	SW846 9040C**	Non-Potable Water
Hexavalent Chromium	SW846 7196A**	Non-Potable Water
Organics		
EDB and DBCP	SW846 8011 Mod**	Solid and Chemical Material
Volatile Organics	SW846 8260B, C**	Solid and Chemical Material
Semi-Volatile Organics	SW846 8270D**	Solid and Chemical Material
Semi-Volatile Organics	SW846 8270D SIM**	Solid and Chemical Material
Gasoline Range Organics	SW846 8015C,D**	Solid and Chemical Material
Diesel Range Organics	SW846 8015C,D**	Solid and Chemical Material
Alcohols	SW846 8015C,D**	Solid and Chemical Material
Polynuclear-Aromatic Hydrocarbons	SW846 8310**	Solid and Chemical Material
Explosives	SW846 8330A**, 8332**	Solid and Chemical Material
Explosives	SW846 8330B**	Solid and Chemical Material
Organochlorine Pesticides	SW846 8081B**	Solid and Chemical Material
Polychlorinated Biphenyls	SW846 8082A**	Solid and Chemical Material
Chlorinated Herbicides	SW846 8151A**	Solid and Chemical Material
Organophosphorus Pesticides	SW846 8141B**	Solid and Chemical Material
Perchlorate	SW-846 6850	Solid and Chemical Material
Perfluorinated Carboxylic Acids and Sulfonates	EPA 537 MOD** (ALS MS014)	Solid and Chemical Material
Total Petroleum Hydrocarbons	FLPRO**	Solid and Chemical Material
Tennessee EPH	TN-EPH**	Solid and Chemical Material

Method Type	Method Number	Regulatory Program
Tennessee GRO	TN-GRO**	Solid and Chemical Material
Wisconsin DRO	WI-DRO**	Solid and Chemical Material
Petroleum Hydrocarbons	Iowa OA-1**	Solid and Chemical Material
Petroleum Hydrocarbons	Iowa OA-2**	Solid and Chemical Material
Volatile Petro. Hydrocarbons	Massachusetts VPH, 2004**	Solid and Chemical Material
Extractable Petro. Hydrocarbons	Massachusetts EPH, 2004**	Solid and Chemical Material
Total Petroleum Hydrocarbons	TX-1005**	Solid and Chemical Material
Acrylamide	SW846 8316	Solid and Chemical Material
Metals		
ICP: General	SW846 6010C**	Solid and Chemical Material
ICP/MS: General	SW846 6020A**	Solid and Chemical Material
Cold Vapor Mercury	SW846 7471B**	Solid and Chemical Material
Inorganic WetChem		
Ion Chromatography (Bromide, Fluoride, Chloride, Sulfate, Nitrite, Nitrate,) – Aqueous	SW846 9056A**	Solid and Chemical Material
Oil & Grease, Gravimetric – Solid	SW846 9071A**	Solid and Chemical Material
Total CN	SW846 9012B**	Solid and Chemical Material
Total Organic Carbon	SW846 9060A**	Solid and Chemical Material
Ammonia	EPA 350.1	Solid and Chemical Material
Total Kjeldahl Nitrogen	EPA 351.2	Solid and Chemical Material
Total Phosphorus	EPA 365.3	Solid and Chemical Material
Waste Ignitability	SW846 1010A**	Solid and Chemical

Method Type	Method Number	Regulatory Program
		Material
Hexavalent Chromium/soils	SW846 7196A**	Solid and Chemical Material
Corrosivity & pH – aqueous	SW846 9040C**	Solid and Chemical Material
Corrosivity & pH – solid	SW846 9045D**	Solid and Chemical Material
Cyanide Reactivity	SW846 Chapter 7**	Solid and Chemical Material
Sulfide Reactivity	SW846 Chapter 7**	Solid and Chemical Material
Organics		
Volatile Organics	TO-3	Air and Emissions
Preparation Methods*		
Liquid/Liquid Extraction, Water	SW846 3510C	
Micro-extraction, Water	SW846 3511	
Solid Phase Extraction, Water	SW846 3535A	
Solids Extraction by Sonication	SW846 3550B	
Microwave-assisted extraction, solids	SW846 3546	
Acid/Base Partitioning	SW846 3650B	
Sulfur Cleanup of Extracts	SW846 3660B	
Sulfuric Acid Cleanup	SW846 3665A	
Purge & Trap - Aqueous	SW846 5030B	
Purge & Trap – Solids	SW846 5035A	
Total Recoverable Metals Digestion	EPA 200.7	
Non-Pot. Water Digest: ICP	SW846 3010A	
Alkaline Digestion of Soils for Hexavalent Chromium	SW846 3060A	
Digestion of Soils for ICP	SW846 3050B	
TCLP	SW846 1311	
SPLP	SW846 1312	

* Preparation methods are not listed on Primary TNI Accreditation per State of Florida DOH rules. However, for the benefit of other accrediting authorities, these methods are inspected during FDOH visits. Listing of surveyed and approved preparation methods is available from on-site inspection report.
** Methods certified by DoD ELAP

Non-TNI-Accredited Fields of Testing

Method Type	Method Number	Regulatory Program
Organics		
Thiodiglycol	SASE in-house method (HPLC)	Non-Potable Water, Solid and Chemical Material
Volatile Petroleum Hydrocarbons	Missouri Gasoline Range Organics	Non-Potable Water, Solid and Chemical Material
Extractable Hydrocarbons	Missouri Diesel Range Organics	Non-Potable Water, Solid and Chemical Material
Extractable Hydrocarbons	Missouri Oil Range Organic	Non-Potable Water, Solid and Chemical Material
Volatile Petroleum Hydrocarbons	Alaska AK-101**	Non-Potable Water, Solid and Chemical Material
Extractable Hydrocarbons	Alaska AK-102**	Non-Potable Water, Solid and Chemical Material
Extractable Hydrocarbons	Alaska AK-103**	Non-Potable Water, Solid and Chemical Material
Volatile Petroleum Hydrocarbons	OK GRO**	Non-Potable Water, Solid and Chemical Material
Extractable Hydrocarbons	OK DRO**	Non-Potable Water, Solid and Chemical Material
Inorganic WetChem		
Oxidation-Reduction Potential	ASTM D1498-76, mod. for solids	Solid and Chemical Material
Percent Ash (dry basis)	ASTM D2974-87, D482-91	Solid and Chemical Material
Grain Size (hydrometer)	ASTM D422-63	Solid and Chemical Material
Sieve Testing	ASTM D422-63	Solid and Chemical

Method Type	Method Number	Regulatory Program
		Material
Specific Gravity	ASTM D1298-85	Solid and Chemical Material
Acidity	SM2310B-11	Non-Potable Water
Dissolved Oxygen	EPA 360.1	Non-Potable Water
Mineral Suspended Solids	EPA 160.2/160.4	Non-Potable Water
Oil & Grease, Gravimetric – AQ	EPA 1664B	Non-Potable Water
Percent Solids	SM2540G-11	Solid and Chemical Material
Settleable Solids	EPA 160.5	Non-Potable Water
Total Mineral Solids	EPA 160.4	Non-Potable Water
Total Residual Chlorine	EPA 330.5	Non-Potable Water
Total Volatile Solids	EPA 160.4	Non-Potable Water
Volatile Suspended Solids	EPA 160.2/160.4	Non-Potable Water
CÑ Amenable to Chlorination	EPA 335.4	Solid and Chemical Material
Bicarbonate, Carbonate, CO2 - calculation	SM2320B-11, SM4500 CO2D-11	Non-Potable Water
Ferrous Iron	SM3500 FE-D-11	Non-Potable Water
Salinity - calculation	SM2520B-11	Non-Potable Water
Paint Filter Test	SW846 9095	Solid and Chemical Material
Corrosivity towards steel	SW846 1110	Solid and Chemical Material
Corrosivity & pH – aqueous	SW846 9040C	Solid and Chemical Material

Appendix III

Equipment List

ORGANIC INSTRUMENTATION

Instrument	Model	Location	Serial #	Year
GC/MS	Agilent 5975TAD MSD/OI 4551/4660	MS-VOA	US13042A19	2013
GC/MS	Agilent 5975TAD MSD/OI 4551/4660	MS-VOA	US11172705	2011
GC/MS	Agilent 5975TAD MSD/OI 4551/4660	MS-VOA	US11322911	2011
GC/MS	Agilent 5975TAD MSD/OI 4551/4660	MS-VOA	US10102029	2010
GC/MS	Agilent 5975TAD MSD/OI 4551/4660	MS-VOA	US83120965	2008
GC/MS	Agilent 5975N MSD/Agilent 7683 AS	SVOC Lab	US71225975	2007
GC/MS	Agilent 5975N MSD/Agilent 7683 AS	SVOC Lab	US62724401	2006
GC/MS	Agilent 5975N MSD/Agilent 7683 AS	SVOC Lab	US53921303	2005
GC/MS	Agilent 5973N MSD/Agilent 7683 AS	SVOC Lab	US40620599	2004
GC/MS	Agilent 5973 MSD/OI 4551/4660	MS-VOA	US41746628	2004
GC/MS	Agilent 5973 MSD/OI 4551/4660	MS-VOA	US41746633	2004
GC/MS	Agilent 5973 MSD/OI 4560/4552 Archon	Soil VOA	US21843765	2002
GC/MS	Agilent 5973 MSD/OI 4551/4660	MS-VOA	US21844034	2002
GC/MS	Agilent 5973 MSD/OI 4660/4552 Archon	Soil VOA	US02440350	2000
GC/MS	Agilent 5973 MSD/OI 4551/4660	MS-VOA	US94240108	1999
GC/MS	Agilent 5973 MSD/Agilent 7683 AS	SVOC Lab	US82311290	1998
GC/MS	Agilent 5973 MSD/Agilent 7683 AS	SVOC Lab	US81211109	1998
GC/MS	Agilent 5973A MSD/OI 4660/4552 Archon	Soil VOA	US82321728	1998
GC/MS	Agilent 5973A MSD/OI 4660/4551	MS-VOA	US63810329	1996
GC	Agilent 7890A/Dual FID/7693 AS	SVOC Lab	CN13161042	2013
GC	Agilent 7890A/Dual FID/7683B AS	SVOC Lab	CN12121006	2012
GC	Agilent 7890A/Dual ECD/7683B AS	SVOC Lab	CN10842133	2008
GC	Agilent 7890A/Dual FID/7693 AS	SVOC Lab	CN10902149	2009
GC	Agilent 7890A/Dual ECD/7683B AS	SVOC Lab	CN10741128	2007
GC	Agilent 6890/Dual FPD/7683B AS	SVOC Lab	US10643024	2006
GC	Agilent 6890/Dual FID/7683B AS	SVOC Lab	CN10641049	2006

Instrument	Model	Location	Serial #	Year
GC	Agilent 6890/Dual ECD/7683B AS	SVOC Lab	CN10641081	2006
GC	Agilent 6890/Dual ECD/7683B AS	SVOC Lab	US10613003	2006
GC	Agilent 6890/PID/PID/OI 4560/4552 Archon	GC VOA	CN10421047	2004
GC	Agilent 6890/PID/FID/ENTECH 7032A-LB	GC VOA	US10239007	2002
GC	Agilent 6890N/Dual FID/HP 7683 AS	SVOC Lab	CN10425061	2004
GC	Agilent 6890N/Dual ECD/HP 7683 AS	SVOC Lab	US10333015	2003
GC	Agilent 6890/Dual ECD/HP 7683 AS	SVOC Lab	US00036916	2000
GC	Agilent 6890/Dual ECD/HP 7683 AS	SVOC Lab	US00028304	1999
GC	Hewlett-Packard 5890/PID/FID/ OI 4560/4552 Archon	GC VOA	3336A60617	1993
GC	Hewlett-Packard 5890/PID/FID/ OI 4560/4552 Archon	Soil VOA	3336A61096	1993
GC	Hewlett-Packard 5890/PID/FID/ OI 4560/4552 Archon	GC VOA	3336A51045	1993
GC	Hewlett-Packard 5890/PID/FID/OI 4560/4552 Archon	GC VOA	3203A41646	1992
GC	Hewlett-Packard 5890/PID/FID/OI 4560/4552 Archon (screening instrument)	GC VOA	3223A42867	1992
GC	Hewlett-Packard 5890/PID/FID OI 4560/4552 Archon	Soil VOA	3029A29748	1990
GC	Hewlett-Packard 5890/FID	GC VOA	2843A20183	1988
GC	Hewlett-Packard 5890/FID	GC VOA	2728A12705	1987
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE91606857	1999
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE23917648	2002
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE01608404	2000
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE40522115	2004
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE03000863	2003
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE61800775	2006

Instrument	Model	Location	Serial #	Year
HPLC	Agilent 1100 Automated LC System	HPLC Room	DE33219455	2003
LC/MS/MS	Agilent 1200/6460 LC/Triple Quad	HPLC Room	SG10447001	2011
O-Prep	ESSA LM2-P Ring and Puck mill	Explosives Prep Lab	215090-004	2008
O-prep	Microwave extractor, 2 units	Organic Prep Lab	Multiple	Various
O-Prep	TurboVap concentrators, 8 units	Organic Prep Lab	Multiple	Various
O-Prep	Buchi solvent recovery system, 3 units	Organic Prep Lab	Multiple	2014
O-Prep	Sonicator 4 units	Organic Prep Lab		Various
O-Prep	N-Vap	Organic Prep Lab	479200-2000	2000
Data System	Hewlett-Packard/MS ChemStation	Labwide		1999, with subsequent upgrades

Inorganic Instrumentation

Instrument	Model	Location	Serial #	Year
ICP/MS	Agilent 7700 Series	Metals Lab	JP12151709	2012
ICP	Thermo ICAP 6000 Series	Metals Lab	20100903	2010
ICP	Thermo ICAP 6000 Series	Metals Lab	20103825	2010
Mercury Analyzer	Leeman Hydra AA II	Metals Lab	2019	2012
Mercury Analyzer	Leeman Hydra AA II	Metals Lab	2004	2012
TOC Analyzer	Shimadzu	WetChem IC room	H51404235007	2004
TOC Analyzer	Shimadzu	WetChem IC room	H51404735099	2010
IC	Dionex IC-2100	WetChem IC room	10110002	2010
IC	Dionex IC-2000	WetChem IC room	04070250	2004
Auto Analyzer	QuickChem 8500 Series	WetChem main room	050500000130	2005
Auto Analyzer	QuickChem 8500 Series 2	WetChem main room	111200001380	2011
Spectrophotometer	Milton-Roy Spectronic 200	WetChem main room	2 units	2000

Instrument	Model	Location	Serial #	Year
Digestion block	DigiPrep	WetChem main room	4 units	2005
Centrifuge	CentraCL2	WetChem main room	42613052	2003
MicroDistillation Block	Lachat	WetChem main room	2 units	2005

LIMS		
Instrument	**Model**	**Year**
LIMS	Stratus Dual Server; Oracle 10G Database	2013

Appendix IV

Certification Summary

Certifying Authority	Certification Program	Registration No.
Alaska	Contaminated Sites	UST-088
Arkansas	Solid/Hazardous Wastes, Non-Potable Water	88-0620
California (NELAP)	Potable Water, Solid/Hazardous Waste	04226CA
Department of Defense (DoD)	Non-Potable Water, Solid and Chemical Materials	L-2229
Florida (NELAP)	Potable, Non-Potable, Solid Waste, UST, Air Toxics	E83510
Georgia	Wastewater/Microbiology analyst	Not Applicable
Illinois	Solid/Hazardous Wastes, Non-Potable Water	
Iowa	UST, Solid/Hazardous Wastes, Non-Potable Water	IA366
Kansas (NELAP)	Solid/Hazardous Wastes, Non-Potable Water	E-10327
Kentucky	Underground Storage Tank Program	0065
Kentucky	Waste Water Program	98023
Louisiana (NELAP)	Solid/Hazardous Wastes	38582
Massachusetts	Non-Potable Water	M-FL946
Mississippi	Potable Water	Not Applicable
Nevada	Non-Potable Water, Solid/Hazardous Wastes	FL009462008A
New Jersey (NELAP)	Solid/Hazardous Wastes, Non-Potable Water	FL002
New York	Solid/Hazardous Wastes, Non-Potable Water	12022
North Carolina	Solid/Hazardous Wastes, Non-Potable Water	573
Oklahoma	Non-Potable Water, Solid/Hazardous Waste	9959
South Carolina	Solid/Hazardous Wastes, Non-Potable Water	96038001
Texas (NELAP)	Non-Potable Water, Solid/Hazardous Waste	T104704040-08-TX
US Dept. of Agriculture	Foreign Soils Permit	P330-16-00126
Utah (NELAP)	Potable, Non-Potable, Solid/Chemical Materials	FL009462008A
Virginia (NELAP)	Potable, Non-Potable, Solid/Chemical Materials	460177
Washington	Potable, Non-Potable, Solid/Chemical Materials, Air	C2046
West Virginia	Solid/Hazardous Wastes, Non-Potable Water	304

Appendix V

SOP List

SOP #	TITLE
	Organic Preparation Department
OP002	SOP for Glassware Cleaning and Storage
OP003	SOP for Reagent Prep
OP006	SOP for the Extraction of Semi-volatile Organics (BNAs) from Aqueous Samples
OP007	SOP for the Extraction of Semi-volatile Organics (BNAs) from Solid Samples
OP007TV	SOP for the Extraction of Semi-volatile Organics (BNAs) from Solid Samples, TurboVap option
OP008	SOP for the Extraction of Pesticides/PCBs from Aqueous Samples
OP009	SOP for the Extraction of Pesticides/PCBs from Solid Samples
OP009MW	SOP for the Extraction of Pesticides/PCBs from Solid Samples, microwave
OP010	SOP for the Extraction of Diesel Range Organics (DRO) from Aqueous Samples
OP011	SOP for the Extraction of Diesel Range Organics (DRO) from Solid Samples
OP011MW	SOP for the Extraction of Diesel Range Organics (DRO) from Solid Samples
OP012	SOP for the Extraction of Petroleum Related Organics (FL-PRO) from Aqueous Samples
OP013	SOP for the Extraction of Petroleum Related Organics (FL-PRO) from Solid Samples
OP014	SOP for the Extraction of PAHs from Aqueous Samples (HPLC)
OP015	SOP for the Extraction of PAHs from Solid Samples (HPLC)
OP016	SOP for the Extraction of EDB/DBCP from Aqueous Samples
OP017	SOP for the Extraction of EDB/DBCP from Solid Samples
OP018	SOP for the Extraction of Explosives from Aqueous Samples, 8330A/B
OP019	SOP for the Extraction of Explosives from Solid Samples, 8330A
OP020	SOP for Sample Introduction via SW846-5035
OP021	SOP for Sample Introduction via SW846-5030B
OP024	Standard Operating Procedure For The Extraction Of Nitroaromatics From Water Samples
OP025	SOP For Sample Preparation For Dissolved Gases In Aqueous Samples
OP026	SOP For The Extraction Of Extractable Petroleum Products (OA-2) From Water Samples
OP027	SOP For The Extraction Of Extractable Petroleum Products (OA-2) From Solid Samples
OP028	SOP For The Extraction Of Diesel And Oil Range Organics From Water Samples
OP029	SOP For The Extraction Of Diesel And Oil Range Organics From Solid Samples
OP030	SOP For The Extraction Of Extractable Petroleum Hydrocarbons From Water Samples (Tennessee EPH)
OP031	SOP For The Extraction Of Extractable Petroleum Hydrocarbons From Solid

SOP #	TITLE
	Samples (Tennessee EPH)
OP032	SOP For The Extraction Of Volatile Petroleum Hydrocarbons From Soil Samples, MA-VPH
OP033	SOP For The Extraction Of PCBs From Wipes
OP034	SOP For The Extraction Of Diesel Range Organics (DRO) From Aqueous Samples WI-DRO
OP035	SOP For The Extraction Of Massachusetts Extractable Petroleum Hydrocarbons From Water Samples
OP036MW	SOP For The Extraction Of Massachusetts Extractable Petroleum Hydrocarbons From Solid Samples, Microwave option
OP037	SOP For The Extraction Of Chlorinated Herbicides From Water Samples
OP038MW	SOP For The Extraction Of Chlorinated Herbicides From Soil Samples, microwave
OP039	SOP For The Solid Phase Extraction (SPE) Cartridge Cleanup Of Pesticide Extracts
OP040	SOP For SPLP Leaching Of SVOC And Metals
OP041	SOP For TCLP Leaching Of VOC
OP042	SOP For SPLP Leaching Of SVOC And Metals
OP043	SOP For SPLP Leaching Of VOC
OP044	SOP For The Extraction Of Organophosphorus Pesticides From Water Samples
OP044SP	SOP For The Extraction Of Organophosphorus Pesticides From Water Samples, Solid Phase Extraction
OP045MW	SOP For The Extraction Of Organophosphorus Pesticides From Soil Samples, microwave
OP046	SOP for the Extraction of Explosives from Solid Samples, SW-8330B
OP048	SOP for the Extraction of PCB Congeners from Aqueous Samples
OP049	SOP for the Extraction of PCB Congeners from Solid Samples
OP050	SOP For The Extraction Of Alaska Extractable Petroleum Hydrocarbons From Water Samples
OP051	SOP For The Extraction Of Alaska Extractable Petroleum Hydrocarbons From Solid Samples
OP052	SOP For The Extraction Of Oklahoma Extractable Petroleum Hydrocarbons From Water Samples
OP053	SOP For The Extraction Of Oklahoma Extractable Petroleum Hydrocarbons From Solid Samples
OP054	SOP For The Extraction Of 1,4-Dioxane From Water Samples
OP055	SOP For The Extraction Of Petroleum Hydrocarbons From Water Samples, TX-1005
OP056	SOP For The Extraction Of Petroleum Hydrocarbons From Solid Samples, TX-1005
OP057	SOP for Sample Introduction via AK-101
OP058	SOP For Extraction Perfluorinated Alkyl Compounds From Water Samples
OP059	SOP for Extraction of PAH and select analytes for 8270 SIM analysis from

SOP #	TITLE
	Aqueous samples
OP060	SOP for Extraction of PAH and select analytes for 8270 SIM analysis from Solid samples
OP061	SOP for Reduced Volume Extraction of PAH from water sample for GC/MS LVI
OP062	SOP for microextraction of PAH from water samples 3511.

Gas Chromatography/ HPLC SOPs

GC002	Analysis Of 1,2-Dibromoethane (EDB) And 1,2-Dibromo-3-Chloropropane (DBCP) By Gas Chromatography, Electron Capture Detector
GC004	Aromatic Volatiles By Gas Chromatography Using PID Detectors EPA 602
GC005	Analysis Of Organochlorine Pesticides By Gas Chromatography, Electron Capture Detector EPA 608
GC006	Analysis Of Polychlorinated Biphenyls By Gas Chromatography, Electron Capture Detector EPA 608
GC007	Analysis Of Polynuclear Aromatic Hydrocarbons By Gas Chromatography, Flame Ionization Detector EPA 610
GC008	Analysis Of Petroleum Range Organics By Gas Chromatography Using Flame Ionization Detector
GC009	Analysis Of 1,2-Dibromoethane (EDB) And 1,2-Dibromo-3-Chloropropane (DBCP) By Gas Chromatography, Electron Capture Detector SW-846 8011
GC010	Analysis Of Gasoline Range Organics By Gas Chromatography Using Flame Ionization Detector
GC011	Analysis Of Diesel Range Organics By Gas Chromatography Using Flame Ionization Detector
GC014	Analysis Of Polychlorinated Biphenyls By Gas Chromatography, Electron Capture Detector SW-846 8082
GC015	Analysis Of Organochlorine Pesticides By Gas Chromatography, Electron Capture Detector SW-846 8081
GC016	Analysis Of Nitroaromatics And Nitramines By HPLC
GC017	Aromatic Volatiles By Gas Chromatography Using PID Detectors SW-8021
GC018	Analysis Of Polynuclear Aromatic Hydrocarbons By HPLC SW-846 8310
GC019	Analysis Of Dissolved Gases By Gas Chromatography, Flame Ionization Detector
GC020	Analysis Of Nitroglycerine And PETN By HPLC
GC021	Analysis Of Volatile Petroleum Hydrocarbons By Gas Chromatography
GC022	Analysis Of Extractable Petroleum Products By Gas Chromatography Using Flame Ionization Detector OA-2
GC023	Analysis Of Diesel And Oil Range Organics By Gas Chromatography Using Flame Ionization Detector
GC024	Analysis Of Petroleum Hydrocarbons By Gas Chromatography Using Flame Ionization Detector (Tennessee EPH)
GC025	Analysis Of Nitroaromatics By Gas Chromatography Using Electron Capture

SOP #	TITLE
	Detector
GC026	Method For Determination Of Volatile Petroleum Hydrocarbons By GC-PID/FID
GC027	Analysis Of Non-Halogenated Organics By Gas Chromatography Using Flame Ionization Detector
GC028	Analysis Of Gasoline Range Organics By Gas Chromatography Using Flame Ionization Detector TDEC GRO
GC029	Analysis Of Diesel Range Organics By Gas Chromatography Using Flame Ionization Detector Wi DRO
GC030	Analysis Of Extractable Petroleum Hydrocarbons By Gas Chromatography Using Flame Ionization Detector MA-EPH
GC031	Analysis Of Chlorinated Herbicides Using GC-ECD
GC032	Analysis Of Organophosphorus Pesticides Using GC-NPD Or FPD
GC033	Air Analysis By GC-PID/FID
GC034	Analysis Of Nitroaromatics, Nitramines And Nitrate Esters By HPLC Method 8330B
GC035	Screening Of Volatile Organics By GC-PID/FID
GC036	Analysis of PCB Congeners by ECD
GC037	Analysis of Diesel and Oil Range Organics by GC/FID, AK-102, AK-103
GC038	Analysis of Gasoline Range Organics by GC/FID, AK-101
GC039	Analysis of Diesel Range Organics by GC/FID, OK-GRO
GC040	Analysis of Gasoline Range Organics by GC/FID, OK-GRO
GC041	Analysis of N-Nitroso-N-Ethylurea by HPLC
GC042	Analysis of Thiodiglycol by HPLC
GC043	Analysis of Acrylamide by HPLC
GC044	Analysis of Petroleum Organics by TX-1005
GC045	Automated Fractionation of MA-EPH extracts
GC046	SOP for Screening Petroleum Range Organics by FID
GC047	Nitroguanidine by HPLC
GC048	Analysis of KS LRH by GC-FID
GC049	Analysis of KS MRH-HPH by GC-FID

Mass-Spectrometry SOPs

MS003	Analysis of Volatile Organics by EPA Method 624
MS004	Analysis of Semi-volatile Organics by EPA Method 625
MS005	Analysis of Volatile Organics by EPA Method 8260B
MS006	Analysis of Semi-volatile Organics by EPA Method 8270C
MS008	Analysis of Semi-volatile Organics by EPA Method 8270C SIM
MS009	Analysis of Volatile Organics by GC/MS
MS010	Analysis of Volatile Organics by GC/MS SIM
MS011	Analysis of Semi-volatile Organics by EPA Method 8270D
MS012	Analysis of 1,4-Dioxane by EPA 522
MS013	Analysis of Perchlorate by SW-846 6850

SOP #	TITLE
MS014	Analysis of PFOS/PFOA by LC/MS/MSD
MS015	Analysis of 8270 SIM LVI
MS016	Analysis of Volatile Organics by EPA Method 8260C
MS017	Analysis of PFOS/PFOA by LC/MS/MSD, method EPA 537, Drinking water
MS018	Analysis of Amines by LC/MS/MSD,

Quality Assurance SOPs

QA001	Preparation, Approval, Distribution & Archiving Of Standard Operating Procedures (SOPs)
QA002	Calibration Of Thermometers
QA003	Personnel Training And Analyst Proficiency
QA004	Temperature Monitoring
QA005	Calibration Of Analytical Balances
QA006	Eppendorf Pipette Calibration
QA007	Sample Batching Procedure
QA008	Creating New Accounts
QA009	Creating New Projects
QA010	Confidentiality Protection Procedures
QA011	Signature Authority
QA012	Employee Technical Ethics Responsibilities
QA013	Client Complaint Resolution Procedure
QA014	Procedures For The Purchase Of Laboratory Supplies
QA015	Procedures For The Preparation, Distribution, Use And Archiving Of Laboratory Logbooks
QA016	Corrective Action Procedure
QA017	Standards Traceability Documentation Procedure
QA018	Procedure For Login, Management, Handling, And Reporting Of Proficiency Test (Pt) Samples
QA019	Quality System Review
QA020	Procedure For Developing Method Performance Criteria And Experimental Method Detection Limits
QA021	Subcontracting Procedures
QA022	Internal Audit Procedure
QA023	Fume Hood Inspection
QA027	Review Of Inorganics Data
QA028	Review Of Organics Data
QA029	Manual Integration Of Chromatographic Peaks
QA030	Procedure For The Development And Use Of in-house Quality Control Criteria
QA031	Air Quality Monitoring Of Extraction Laboratory
QA032	Routine Maintenance For Major Analytical Instrumentation
QA033	Laboratory Safety
QA034	Sample Homogenizing

SOP #	TITLE
QA035	Solvent Testing And Approval
QA036	Data Package Generation
QA037	Deionized Water Quality Control Procedure
QA038	Data Integrity Training Procedure
QA039	Data Integrity Monitoring Procedure
QA040	Procedure For Conducting Data Integrity Investigations
QA041	Procedure For The Confidential Reporting Of Data Integrity Issues
QA042	Basic Calculations For General Chemistry Methods
QA043	Data Qualifier SOP
QA044	Calibration Of Micro-Distillation Tubes
QA045	Estimation of Uncertainty
QA046	Document Control
QA047	Management of Client Project
QA048	Data Entry for Log-In

General Chemistry SOPs

GNSOP: 101	Acidity (pH 8.2)
GNSOP: 102	Alkalinity, Total (pH 4.5)
GNSOP: 103	Ammonia – Distillation Procedure
GNSOP: 104	Nitrogen, Ammonia
GNSOP: 105	Bicarbonate, Carbonate, Free Carbon Dioxide
GNSOP: 106	Chemical Oxygen Demand
GNSOP: 107	Chloride by Titration
GNSOP: 109	Color, Apparent
GNSOP: 110	Chromium, Hexavalent (Water)
GNSOP: 113	Cyanide Distillation/Aqueous And Solid Samples
GNSOP: 115	Cyanide, Total
GNSOP: 116	Dissolved Oxygen
GNSOP: 121	Ignitability
GNSOP: 122	Anionic Surfactants As MBAS
GNSOP: 123	Nitrogen, Nitrite
GNSOP: 126	Ortho Phosphate
GNSOP: 127	Paint Filter Liquids Test
GNSOP: 128	Phenols Distillation, Soil And Water Samples
GNSOP: 130	Phenols, Total Recoverable
GNSOP: 133	Settleable Solids
GNSOP: 134	Total Suspended Solids (Non Filterable Residue)
GNSOP: 135	Total Dissolved Solids (Total Filterable Residue)
GNSOP: 136	Reactive Sulfide And Reactive Cyanide
GNSOP: 137	pH By Electrode - Water
GNSOP: 140	Sulfide
GNSOP: 144	Total Phosphorus
GNSOP: 145	Turbidity

SOP #	TITLE
GNSOP: 147	Winkler Titration For DO Standardization
GNSOP: 161	Percent Solids
GNSOP: 163	Specific Conductance At 25 C.
GNSOP: 166	pH By Electrode – Soil
GNSOP: 167	Biochemical Oxygen Demand (BOD)
GNSOP: 171	Hexachromium In Soils
GNSOP: 179	Corrosivity (Soil pH By Electrode)
GNSOP: 182	Total Kjeldahl Nitrogen
GNSOP: 189	Corrosivity Toward Steel
GNSOP: 190	Total Nitrogen, Organic Nitrogen
GNSOP: 191	Nitrogen, Nitrate
GNSOP: 192	Carbonaceous Biochemical Oxygen Demand (CBOD)
GNSOP: 193	Oxidation-Reduction Potential
GNSOP: 194	Ferrous Iron
GNSOP: 196	Glassware Cleaning
GNSOP: 197	Anions By Ion Chromatography
GNSOP: 211	Oil & Grease And PHC By 1664
GNSOP: 212	Fractional Organic Carbon
GNSOP: 213	Walkley-Black Total Organic Carbon
GNSOP: 214	Particle Size By Sieve
GNSOP: 215	TOC In Water
GNSOP: 216	Particle Size By Hydrometer
GNSOP: 218	Perchlorate
GNSOP: 219	Bulk Density
GNSOP: 222	Un-Ionized Ammonia Calculation
GNSOP: 224	Hardness By Calculation
GNSOP: 225	Cation Exchange Capacity Of Soils (Sodium Acetate)
GNSOP: 226	TOC In Soil
GNSOP: 227	Oil And Grease – Gravimetric Analysis (Soils)
GNSOP: 228	Anions By Ion Chromatography - IC 2000
GNSOP: 231	% Ash
GNSOP: 232	Determination Of Nitrate and Nitrite by Lachat
GNSOP: 233	Sulfite
GNSOP: 234	Total Solids, Gravimetric
GNSOP: 235	Total Volatile Solids, Gravimetric

Metals SOPs

MET 100	Metals By Inductively Coupled Plasma, EPA 6010C
MET 103	Digestion Of Water Samples For Flame And ICP Analysis
MET 104	Digestion Of Soils For ICP Analysis
MET 105	Cold Vapor Analysis Of Mercury For Soils
MET 106	Cold Vapor Analysis Of Mercury For Water Samples
MET 107	Metals By Inductively Coupled Plasma, Mass-Spectrometry

SOP #	TITLE
MET 108	Metals By Inductively Coupled Plasma, EPA 6010D

Sample Management SOPs

SAM101	Sample Receipt And Storage
SAM102	Procedure For Sample Bottle Preparation And Shipment
SAM104	Sample Container Quality Control
SAM108	Sample And Laboratory Waste Disposition
SAM109	Foreign Soil receipt and Handling

Appendix VI

Data Integrity Training Acknowledgement and Ethical Conduct Agreement

I understand that SGS Accutest is committed to having its employees perform their duties ethically and responsibly. By signing this document, I agree to uphold SGS Accutest commitment to ethics and integrity as follows:

I. I understand the high ethical standards required of me with regard to the duties I perform and the data I report in connection with my employment at SGS Accutest.

II. I have received formal instruction on the code of ethics that has been adopted by SGS Accutest during my orientation and agree to comply with these requirements.

III. I have received formal instruction on the elements of SGS Accutest's Data Integrity Policy and have been informed of the following specific procedures:

- *a. Formal procedures for the confidential reporting of data integrity issues are available, which can be used by any employee,*
- *b. A data integrity investigation is conducted when data issues are identified that may negatively impact data integrity.*
- *c. Routine data integrity monitoring is conducted on sample data, which may include an evaluation of the data I produce,*

IV. I have attended the Data Integrity training detailing SGS Accutest Data Integrity and Ethics Program as required.

V. I am aware that data fraud is a punishable crime that may include fines and/or imprisonment upon conviction.

VI. I also agree to the following:

- *a. I shall not intentionally report data values, which are not the actual values observed or measured.*
- *b. I shall not intentionally modify data values unless the modification can be technically justified through a measurable analytical process.*
- *c. I shall not intentionally report dates and times of data analysis that are not the true and actual times the data analysis was conducted.*
- *d. I shall not condone any accidental or intentional reporting of inauthentic data by other employees and immediately report it's occurrence to my superiors.*
- *e. I shall immediately report any accidental reporting of inauthentic data by myself to my superiors.*
- *f. I will, at all times, handle client samples and SGS Accutest instrumentation as required by the SGS Accutest Standard Operating Procedures.*

g. *I will not intentionally deviate from, or fail to follow, the SGS Accutest Standard Operating Procedures at any time except as authorized by this document.*

h. *I understand that deviations from a Standard Operating procedure are allowed only when the deviations are clearly presented in writing by supervisory, managerial or director level staff and when those deviations do not contradict any part of the SGS Accutest ethics policy. No other personnel are allowed to approve Standard Operating Procedure deviations.*

i. *Anytime someone suggests, recommends, or requests that I do not follow an SGS Accutest Standard Operating Procedure, other than as noted in h above, I shall immediately notify my supervisor, manager, a Quality Assurance Officer, the Lab Director, or the Director of Human Resources.*

j. *Anytime I am uncomfortable or unsure about an action that I am requested to perform, I shall immediately notify my supervisor, manager, a Quality Assurance officer, the Lab Director, or the Director of Human Resources. By doing so, I understand that I will not be punished or penalized for asking for guidance or reporting potential wrongdoing.*

k. *If I intentionally disregard the SGS Accutest Standard Operating Procedures without written authorization to do so, I may face disciplinary action up to and including termination of my employment. Note: unintentional deviation from a Standard Operating Procedure must be documented on discovery and appropriate corrective actions followed.*

l. *If I become aware of another person who appears to be disregarding the SGS Accutest Standard Operating Procedures without written authorization to do so, I shall immediately report it to my supervisor, manager, a Quality Assurance Officer, the Lab Director, or the Director of Human Resources. By failing to do so, I may face disciplinary action up to and including termination of my employment.*

m. *I am aware that intentionally failing to follow an SGS Accutest Standard Operating Procedure, other than as noted in h above, may be illegal and could be considered data fraud. In addition, providing instruction to another person to deviate from a Standard Operation, other than as noted in c above, may be illegal and could be considered data fraud.*

n. *I am aware that data fraud is a crime and is punishable by fines and/or imprisonment upon conviction. It is the general policy of SGS Accutest to cooperate with law enforcement authorities in the investigation and prosecution of such matters.*

o. *I understand that SGS Accutest strictly prohibits unlawful retaliation and I understand that, if I report a violation of the SGS Accutest Standard Operating Procedures or an instruction that would violate the SGS Accutest Standard Operating Procedures, I will not be subjected in any way to any adverse employment action because of my report. I agree that if I believe I am being, or have been, subjected to an adverse employment action because of my report, then I will immediately notify my supervisor, manager, a Quality Assurance officer, the Lab Director, or the Director of Human Resources. I agree that SGS Accutest cannot address or correct any such retaliatory behavior unless it is reported and SGS Accutest is given an opportunity to address or correct such behavior.*

Printed Name	Signature	Date

ANALYSIS OF NITROAROMATICS, NITRAMINES, AND NITRATE ESTERS BY HPLC METHOD SW-846 8330B

Prepared by: Norm Farmer Date: 08/21/17

Approved by: Mike Eger Date: 08/23/17

Annual Review

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

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TITLE: ANALYSIS OF NITROAROMATICS, NITRAMINES, AND NITRATE ESTERS BY HPLC METHOD SW-846 8330B

REFERENCES: SW846 8330B

REVISED SECTIONS: 1.1.3-1.1.7, 1.2.3, 4.5, 6.1, 6.2, 7.1.3, 7.2.1-7.2.3, 7.3.2, 7.4.1.1, 7.4.2, 8.0, 9.2.1, 9.3.1, 9.8.1, 11.1 and 12.0

1.0 SUMMARY, SCOPE AND APPLICATION

1.1 Scope and Application

1.1.1 This method is used to determine the concentrations of specific nitroaromatics, nitramines, and nitrate esters in water and solid matrices utilizing an HPLC equipped with a diode array detector.

1.1.2 The following compounds can be reported by this method:

Octahydro-1,3,5,7-tetranitro-1,3,5,7-tetrazocine (HMX)	
Hexahydro-1,3,5-trinitro-1,3,5-triazine (RDX)	
Methyl-2,4,6-trinitrophenylnitramine (Tetryl)	
1,3-Dinitrobenzene	1,3,5-Trinitrobenzene
2,6-Dinitrotoluene	2,4,6-Trinitrotoluene
2,4-Dinitrotoluene	2,4-diamino-6-Nitrotoluene
2-amino-4,6-Dinitrotoluene	2,6-diamino-4-Nitrotoluene
4-amino-2,6-Dinitrotoluene	2-amino-6-Nitrotoluene
Nitrobenzene	2-amino-4-Nitrotoluene
o-Nitrotoluene	4-amino-2-Nitrotoluene
m-Nitrotoluene	2,4-Diaminotoluene
p-Nitrotoluene	2,6-Diaminotoluene
DNX	3,5-Dinitroaniline
MNX	Nitroglycerine
TNX	PETN

1.1.3 The Lower Limit of Quantitation (LLOQ) or Reporting limits (RL) are based on the extraction procedure and the lowest calibration standard. LLOQs may vary depending on matrix complications and volumes. LLOQs for this method are in the range of 0.2 to 2.0 ug/l for aqueous samples and 100 to 1000 ug/kg for solid samples.

1.1.4 The Method Detection Limit (MDL) for each analyte is evaluated on an annual basis for each matrix and instrument. MDLs are pooled for each matrix, and the final pooled MDLs are verified. The verified MDLs are stored in the LIMS and should be at least 2 to 3 times lower than the LLOQ. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the

reported LLOQ. Note: MDL verifications for 8330B soils must be spiked prior to grinding.

1.1.5 The LLOQ for each analyte is evaluated on an annual basis for each matrix and instrument. The LLOQ verifications are prepared by spiking a clean matrix at 0.5 to 2 times the current LLOQ level. This LLOQ verification is carried through the same preparation and analytical procedures as the samples. Recovery of the analytes should be within the established limits. The DOD QSM requirements for Limit of Detection (LOD) and Limit of Quantitation (LOQ) verifications are different. See SOP QA020 for complete requirements for MDL, LOD, LOQ, and LLOQ.

1.1.6 Compounds detected at concentrations between the LLOQ and MDL are quantitated and qualified as estimated values and reported with either a “J” or “I” qualifier. Some program or project specifications may require that no values below the LLOQ be reported

1.1.7 For DOD projects refer to QSM 4.2, Table F3; QSM 5.0, Table 3; or QSM 5.1 Table B-3 for additional method requirements and data qualifying guidance.

1.2 Summary

1.2.1 This method is adapted from SW846 Method 8330B.

1.2.2 Samples are received, stored and extracted within the appropriate holding times.

1.2.3 Sample preparation is performed in accordance with SGS Accutest – Orlando SOP OP018 and OP046.

1.2.4 The extracts are analyzed on an HPLC equipped with a diode array detector.

1.2.5 Manual integrations are performed in accordance with SOP QA029.

2.0 PRESERVATION AND HOLDING TIME

2.1 Preservation

2.1.1 Aqueous samples shall be collected in amber glass bottles with Teflon lined caps. One-liter or 250ml bottles are recommended for aqueous samples.

2.1.2 Soil samples shall be collected by multi-incremental sampling or by the collection of large volume discreet samples. This can result in sample sizes of one to two kilograms. Samples shall be collected in heavy duty 1 or 2 gallon ziplock bags. It is recommended that the samples be double bagged to prevent punctures.

2.1.3 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until extraction. Soil samples can be stored at room temperature after they have been air dried. The extracts must be stored at ≤ 6°C until analysis.

2.2 Holding Time

2.2.1 Aqueous samples must be extracted within 7 days of collection.

2.2.2 Solid and waste samples must be extracted within 14 days of collection.

2.2.3 Extracts should be analyzed as soon as possible but must be analyzed within 40 days of extraction.

3.0 INTERFERENCES

3.1 Data from all blanks, samples, and spikes must be evaluated for interferences.

3.2 Method interferences may be caused by contaminants in solvents, reagents, or glassware. All of these materials must be demonstrated to be free from interferences.

3.3 Tetryl decomposes rapidly in methanol/water solutions, as well as with heat. All aqueous samples expected to contain tetryl should be diluted with acetonitrile prior to filtration and acidified to pH < 3. Samples and extracts should not be exposed to temperatures above room temperature.

3.4 High levels of 4-amino-2-nitrotoluene may interfere with the surrogate 3,4-dinitrotoluene on the (Zorbax Extend C-18) primary column. In such instances, the surrogate recovery should be calculated from the confirmation column.

3.5 3,5-dinitroaniline partially co-elutes with o-nitrotoluene and p-nitrotoluene on the confirmation column.

3.6 When analyzing the RDX breakdown analytes, TNX may partially coelute with HMX on the primary column and DNX may partially co-elute with HMX on the confirmation columns.

3.7 Samples from sites using alkaline hydrolysis to remediate explosives may have a high basic pH. These samples and extracts should be neutralized prior to analysis to prevent damage to the analytical column.

4.0 DEFINITIONS

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. For all GC and HPLC methods, a CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

4.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

4.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the LLOQ.

4.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

4.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.10 Proficiency Test Sample (PT): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The PT sample is generally prepared by an outside vendor. This method requires that the PT sample go through the entire preparatory procedure including sieving and grinding. PT sample recoveries are used to document laboratory and method performance.

4.11 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.12 Sample Triplicate (TRP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.13 Grinding Blank (GB): An aliquot of blank sand that is processed through the ring and puck mill between different samples. It is used to monitor for carry over between samples ground with the same bowl set.

4.14 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.15 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 REAGENTS

5.1 Water – HPLC grade or equivalent

5.2 Acetonitrile – HPLC grade or equivalent

5.3 Methanol – HPLC grade or equivalent

5.4 Explosives stock standards – Traceable to Certificate of Analysis

5.5 Surrogate standards – 3,4-Dinitrotoluene

6.0 APPARATUS

6.1 HPLC – Agilent Technologies 1100 or 1260

Suitable HPLC equipped with an autosampler, pump, and diode array detector.

Autosampler allows for unattended sample and standard injection throughout the analytical run.

6.2 Data System – Agilent Technologies LC Chemstation rev. A 10.01 or B 04.03
Agilent Technologies MS Chemstation rev. DA 00.01 or EA 02.01

6.2.1 A computer system interfaced to the HPLC that allows for the continuous acquisition and storage of all data obtained throughout the duration of the chromatographic program.

6.2.2 The software must allow for quantitation at multiple wavelengths. Additionally the software should allow for the viewing of the entire UV Spectra acquired over the analytical run. Comparisons can then be made between spectra from standards and samples.

6.2.3 Data is archived to a backup server for long term storage.

6.3 Primary Column – Zorbax Extend C-18 3.5u – 4.6mm X 100mm or equivalent

6.4 Confirmation Column– Zorbax Bonus RP (amine bonded C-18) 5u - 4.6mm X 250mm or equivalent

6.5 Gas-tight syringes, syringe filters, and class "A" volumetric glassware for dilutions of standards and extracts.

7.0 PROCEDURE

7.1 Standards Preparation

Standards are prepared from commercially available certified reference standards. All standards must be logged in the HPLC Standards Logbook. All standards shall be traceable to their original source. The standards should be stored at ≤ 6°C, or as recommended by the manufacturer. Calibration levels, spike and surrogate concentrations, preparation information, and vendor part numbers can be found in the HPLC STD Summary in the Active SOP directory.

7.1.1 Stock Standard Solutions

Stock standards are available from several commercial vendors. All vendors must supply a "Certificate of Analysis" with the standard. The certificate will be retained by the lab. Hold time for unopened stock standards is until the vendor's expiration date. Once opened, the hold time is reduced to one year or the vendor's expiration date (whichever is shorter).

7.1.2 Intermediate Standard Solutions

Intermediate standards are prepared by quantitative dilution of the stock standard with acetonitrile. The hold time for intermediate standards is six months or the vendor's expiration date (whichever is shorter). Intermediate standards may need to be remade if comparison to other standards indicate analyte degradation or concentration changes.

7.1.3 Calibration Standards

Calibration standards for the explosives are prepared at a minimum of five concentration levels through quantitative dilutions of the intermediate standard. Calibration standards are prepared in 75/25 (v/v) water/acetonitrile. The low standard is at a concentration at or below the LLOQ and the remaining standards define the working range of the detector.

Calibration standard concentrations are verified by the analysis of an initial calibration verification (ICV) standard.

7.2 HPLC Conditions

7.2.1 HPLC-BB/PP/II Conditions - Primary Column – (Extend C-18)

100 ul autosampler injection

Mobile phase – Gradient: Water (A), Methanol (B)

Time (min)	Solvent A	Solvent B
0-1.5	79%	21%
1.5-5.8	72%	28%
5.8-11.0	72%	28%
11.0-16.0	65%	35%
16.0-19.0	50%	50%
19.0-23.0	50%	50%

Column temperature – 43.0 °C Constant Flow – 2.0 ml/min

Diode Array Detector – Set to acquire and process data at 254-nm wavelengths using a 10-nm bandwidth. Secondary wavelength may be set to 214-nm. The 254-nm wavelength switches to 270-nm just prior to the elution of the nitrotoluenes. All data from 200-nm to 450-nm wavelengths is stored for spectral evaluation.

HPLC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.2.2 HPLC-GG Conditions - Confirmation Column – (Bonus RP)

100 ul autosampler injection

Mobile phase – Gradient: Water (A), Methanol (B)

Time (min)	Solvent A	Solvent B
0-1.00	41%	59%
1.00-1.25	50%	50%
1.25-17.0	47%	53%
17.0-19.5	41%	59%
19.5-27.0	41%	59%

Column temperature – 23.0 °C Constant Flow – 0.75 ml/min

Diode Array Detector – Set to acquire and process data at 254-nm wavelengths using a 10-nm bandwidth. Secondary wavelength may be set to 214-nm. All data from 200-nm to 450-nm wavelengths is stored for spectral evaluation.

HPLC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.2.3 HPLC-PP Conditions - Confirmation Column – (Bonus RP)

100 ul autosampler injection

Mobile phase – Gradient: Water (A), Methanol (B)

Time (min)	Solvent A	Solvent B
0-1.00	41%	59%
1.00-1.25	50%	50%
1.25-17.0	47%	53%
17.0-19.5	41%	59%
19.5-27.0	41%	59%

Column temperature – 22.0 °C Constant Flow – 0.8 ml/min

Diode Array Detector – Set to acquire and process data at 254-nm wavelengths using a 10-nm bandwidth. Secondary wavelength may be set to 214-nm. All data from 200-nm to 450-nm wavelengths is stored for spectral evaluation.

HPLC conditions are optimized for each instrument. Actual conditions may vary slightly from those listed above.

7.3. Sample Preparation

7.3.1 Water Samples (extracted)

A 250ml or 1000ml aliquot of sample is extracted utilizing a solid phase cartridge. The cartridge is eluted with acetonitrile. The final volume is then adjusted to 2.0ml or 10ml with reagent water.

7.3.2 Solid Samples

A 10-gram aliquot of sample is extracted with 20ml of acetonitrile utilizing a platform shaker. The final volume is then adjusted to 50ml with reagent water. The extract is filtered through a .45um Teflon syringe filter to remove any particulate.

7.4. HPLC Analysis

Instrument calibration consists of two major sections:

Initial Calibration Procedures
Continuing Calibration Verification

7.4.1 Initial Calibration Procedures

Before samples can be run, the HPLC system must be calibrated, and retention time windows must be determined.

7.4.1.1 External Standard Calibration

A minimum 5-point calibration curve is created for the explosives and surrogates. SGS Accutest Laboratories routinely performs a 7-point calibration to maximize the calibration range.

The low point may be omitted from the calibration table for any compound with an RL set at the level two standard. Additionally the high point may be omitted for any compound that exhibits poor linearity at the upper end of the calibration range.

An entire level may be omitted provided that a minimum of 5 points remain. There must be technical justification to omit an entire level. This should be documented in the run log.

Historically, many analytical methods have relied on linear models of the calibration relationship, where the instrument response is directly proportional to the amount of a target compound. The linear model has many advantages including simplicity and ease of use. However, given the advent of new detection techniques and because many methods cannot be optimized for all the analytes to which they may be applied, the analyst is increasingly likely to encounter situations where the linear model neither applies nor is appropriate. The option of using non-linear calibration may be necessary to address specific instrumental techniques. However, it is not EPA's intent to allow non-linear calibration to compensate for detector saturation or avoid proper instrument maintenance.

NOTE: Because of this concern, select programs including SC DHEC do not support the use of non-linear regressions.

Calibration factors (CF) for the explosives and surrogates are determined at each concentration by dividing the area of each compound by the concentration of the standard.

The mean CF and standard deviation of the CF are determined for each analyte. The percent relative standard deviation (%RSD) of the response factors is calculated for each analyte as follows:

%RSD = (Standard Deviation of CF X 100) / Mean CF

If the %RSD $\leq$ 15%, linearity through the origin can be assumed and the mean CF can be used to quantitate target analytes in the samples. Alternatively a calibration curve of response vs. amount can be plotted. This method allows for the use of average response factors, linear regressions, and non-linear regressions. Linear regressions may be unweighted or weighted as $1/x$ or $1/x^2$. If the correlation coefficient (r) is $\geq$0.995 (r^2 $\geq$0.990) then the curve can be used to quantitate target analytes in the samples. Regardless of which calibration model is chosen, the laboratory should visually inspect the curve plots to see how the individual calibration points compare to the plot.

Alternatively either of the two techniques described below may be used to determine whether the calibration function meets acceptable criteria. These involve refitting the calibration data back to the model. Both %

Error and Relative Standard Error (RSE) evaluate the difference between the measured and the true amounts or concentrations used to create the model.

Calculation of the % Error

% ERR = (xi-x'i) / xi * 100

x'i = Measured amount of analyte at calibration level i, in mass or concentration units.

xi = True amount of analyte at calibration level i, in mass or concentration units.

Percent error between the calculated and expected amounts of an analyte should be ≤ 30% for all standards. For some data uses, ≤50% may be acceptable for the lowest calibration point.

Calculation of Relative Standard Error (%RSE)

$$RSE = 100 \times \sqrt{\sum_{i=1}^{n} \left[\frac{x_i' - x_i}{x_i}\right]^2 \Big/ (n-p)}$$

x'i = Measured amount of analyte at calibration level i, in mass or concentration units.

xi = True amount of analyte at calibration level i, in mass or concentration units.

p = Number of terms in the fitting equation.
(average = 1, linear = 2, quadratic = 3)

n = Number of calibration points.

The %RSE acceptance limit criterion is ≤15%.

7.4.1.2 Initial Calibration Verification (ICV)

The validity of the initial calibration curve must be verified through the analysis of an initial calibration verification (ICV) standard. The ICV should be prepared from a second source at a mid-range concentration.

The %D for all analytes of interest should be ≤ 20%. If the ICV does not meet this criteria, a second standard should be prepared. If the ICV still

does not meet criteria, analyze an ICV prepared from a third source. If this ICV meets criteria, proceed with sample analysis.

For any DoD QSM project, if samples must be analyzed with a target analyte having a %D > 20%, then the data must be qualified accordingly.

If the ICV still does not meet criteria, determine which two standards agree. Make fresh calibration standards and an ICV from the two sources that agree. Recalibrate the instrument.

NOTE: Second source standards may not be available for TNX, DNX, and MNX.

7.4.1.3 Retention Time Windows

Retention time windows must be established whenever a new column is installed in an instrument or whenever a major change has been made to an instrument.

Retention time windows are crucial to the identification of target compounds. Absolute retention times are used for compound identification in all GC and HPLC methods that do not employ internal standard calibration. Retention time windows are established to compensate for minor shifts in absolute retention times that result from normal chromatographic variability. The width of the retention time window should be carefully established to minimize the occurrence of both false positive and false negative results.

Retention time windows are established by injecting all standard mixes three times over the course of 72 hours. The width of the retention time window for each analyte, surrogate, and major constituent in multi-component analytes is defined as ± 3 times the standard deviation of the mean absolute retention time or 0.03 minutes, whichever is greater.

Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration.

Peak identification is based on the retention time of a peak falling within the retention time window for a given analyte. Time reference peaks (surrogates) are used to correct for run-to-run variations in retention times due to temperature, flow, or injector fluctuations. HPLC retention times tend to shift more than GC retention times.

The retention time windows should be used as a guide for identifying compounds; however, the experience of the analyst should weigh

heavily in the interpretation of the chromatograms. The analyst should monitor the retention times of known peaks (standards and surrogates) throughout an instrument run as an indication of instrument performance.

Because calculated retention time windows are generally very tight (less than ± 0.03 minutes), the retention time windows for the data processing method are generally set wider than the calculated window. This is done to ensure that the software does not miss any potential "hits". The analyst will then review these "hits" and determine if the retention times are close enough to the retention time of the target analyte to positively identify the peak or to require confirmation.

7.4.2 Continuing Calibration Verification (CCV)

Continuing calibration verification standards for the explosives are prepared at various concentrations; at least one CCV must be below the mid-point of the calibration curve. A continuing calibration standard must be analyzed at the beginning and end of each run to verify that the initial calibration is still valid. Additionally a CCV must be analyzed after every 10 samples.

The percent difference (%D) for each analyte of interest will be monitored. The |%D| should be ≤ 20% for each analyte.

If the first continuing calibration verification does not meet criteria, a second standard may be injected. If the second standard does not meet criteria, the system must be recalibrated. If the second standard meets criteria then the system is considered in control and results may be reported.

Rationale for second standard such as instrument maintenance, clipped column, remade standard, etc should be documented in the run log or maintenance log. Reanalysis of second standard without valid rationale may require the analysis of a third standard (in which case both the second and third standard would have to pass).

NOTE: For any DoD QSM project, if the second standard meets criteria, then a third standard must be analyzed. If the third standard also meets criteria then the system is considered in control and results may be reported.

If the |%D| is greater than 20%, then documented corrective action is necessary. This may include recalibrating the instrument and reanalyzing the samples, performing instrument maintenance to correct the problem and reanalyzing the samples, or qualifying the data. Under certain circumstances, the data may be reportable. i.e. The CCV failed high, the associated QC passed, and the samples were ND.

NOTE: For any DoD QSM project, if samples must be reported with a target analyte having a %D > 20%, then the data must be qualified accordingly, regardless of whether the analyte was detected or not.

NOTE: Any target analytes that are detected in the samples must be bracketed by an acceptable initial calibration curve and acceptable CCV standards; otherwise, the samples must be reanalyzed or the data must be qualified.

7.4.3 Sample Extract Analysis

7.4.3.1 Samples are analyzed in a set referred to as an analysis sequence or batch. A batch consists of the following:

Initial Calibration Standards (or Initial CCV)
QC Extracts
Sample Extracts
CCV Standards

7.4.3.2 One hundred microliters (same amount as standards) of extract is injected into the HPLC by the autosampler. The data system then records the resultant peak responses and retention times.

7.4.3.3 Tentative identification of an analyte occurs when the peaks from the sample extract fall within the established retention time windows for a calibrated compound.

7.4.3.4 The diode array detector is capable of spectral evaluation; however, the UV spectra for some explosives analytes are not very unique. Confirmation by reanalysis on a dissimilar column is required for positive identification.

7.4.3.5 If the peaks of interest fall within the retention time windows on the confirmation column, the identification is confirmed. Quantitation of the analyte on the primary and confirmation column should agree within 40%. If the difference is greater than 40% and no obvious reason can be found, the higher result should be reported and flagged as “estimated”; otherwise, the result from the primary column should be reported.

If doubt over the identity of the analyte exists after primary and confirmation analysis, it may be appropriate to report the analyte as non-detect with an elevated RL and MDL. This should be discussed with the client and documented on the result page.

7.4.3.6 If the compound identification does not confirm, then the result should be reported as ND or “U”.

7.4.3.7 If the analyte response exceeds the linear range of the system, the extract must be diluted and reanalyzed. It is recommended that extracts be diluted so that the response falls into the middle of the calibration curve.

7.4.3.8 If peak identification is prevented by the presence of interferences, further cleanup may be required or the extract must be diluted so that the interference does not mask any analytes. Analysis on the confirmation column may also be beneficial.

7.5. Maintenance and Trouble Shooting

7.5.1 Refer to SOP GC001 for routine instrument maintenance and trouble shooting.

7.5.2 All instrument maintenance must be documented in the appropriate "Instrument Repair and Maintenance" log. The log will include such items as problem, action taken, correction verification, date, and analyst.

7.5.3 Repairs performed by outside vendors must also be documented in the log. The analyst or Department Supervisor responsible for the instrument must complete the log if the repair technician does not.

7.5.4 PC and software changes must be documented in the "Instrument Repair and Maintenance" log. Software changes may require additional validation.

8.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to statistically generated control limits. These control limits are reviewed and updated annually. Control limits are stored in the LIMS. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

DoD QSM projects require the analysis of a sample triplicate (TRP), a proficiency test sample (PT) and equipment grinding blanks (GB).

9.0 QUALITY ASSURANCE / QUALITY CONTROL

Accuracy and matrix bias are monitored by the use of surrogates and by the analysis of a QC set that is prepared with each batch (maximum of 20 samples) of samples. The QC set consists of a method blank (MB), blank spike (BS), matrix spike (MS), matrix spike duplicate (MSD), and sample duplicate (DUP). DoD QSM projects require the analysis of a sample triplicate (TRP), a proficiency test sample (PT) and equipment grinding blanks (GB).

9.1 Surrogates

9.1.1 3,4-Dinitrotoluene is used as the surrogate standard to monitor the efficiency of the extraction.

A known amount of surrogate standard is added to each sample including the QC set prior to extraction. The percent recovery for each surrogate is calculated as follows:

% Recovery = (Sample Amount / Amount Spiked) X 100

The percent recovery must fall within the established control limits for the results to be acceptable.

9.1.2 If the surrogate recovery is not within the established control limits, the following are required.

9.1.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or surrogate solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.1.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.1.2.3 If no problem is found, re-extract and reanalyze the sample. **NOTE:** If the recoveries are high and the sample is non-detect, then re-extraction may not be necessary. For any DoD QSM projects the resulting data must be qualified accordingly. If there is insufficient sample for re-extraction, reanalyze the sample and footnote this on the report.

9.1.2.4 If upon reanalysis, the recovery is still not within control limits, the problem is considered matrix interference. Surrogates from both sets of analysis should be reported on the final report.

9.2 Method Blank

9.2.1 The method blank is either de-ionized water or cleaned sand (depending upon sample matrix) to which the surrogate standard has been added. The method blank is then extracted and taken through all cleanup procedures along with the other samples to determine any contamination from reagents, glassware, or high level samples. The method blank must be free of any analytes of interest or interferences at ½ the required LLOQ to be acceptable. If the method blank is not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated method blank shall be

evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-extracting and reanalyzing the samples or qualifying the results with a "B" or "V" qualifier.

9.2.2 If the MB is contaminated but the samples are non-detect, then the source of contamination should be investigated and documented. The sample results can be reported without qualification.

9.2.3 If the MB is contaminated but the samples results are > 10 times the contamination level, the source of the contamination should be investigated and documented. The samples results may be reported with the appropriate "B" or "V" qualifier. This must be approved by the department supervisor.

9.2.4 If the MB is contaminated but the samples results are < 10 times the contamination level, the source of the contamination should be investigated and documented. The samples should be re-extracted and reanalyzed for confirmation. If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.3 Blank Spike

9.3.1 The blank spike is either de-ionized water or cleaned sand (depending upon sample matrix) to which the surrogate standard and spike standard have been added. The blank spike is then extracted and taken through all cleanup procedures along with the other samples to monitor the efficiency of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (Blank Spike Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the established control limits for the results to be acceptable. The large number of analytes in this method presents a substantial probability that a few of the analytes will fall outside of the established control limits. This may not indicate that the system is out of control; therefore, corrective action may not be necessary.

Upper and lower marginal exceedance (ME) limits can be established to determine when corrective action is necessary. A marginal exceedance in the Blank Spike is defined as a recovery being outside of 3 standard deviations but within 4 standard deviations of the mean.

The number of allowable marginal exceedances is based on the number of analytes in the Blank Spike. Marginal Exceedances must be random. If the same analyte exceeds the BS control limits repeatedly, it is an indication of a systematic problem and corrective action must be taken.

Marginal exceedances are not permitted for analytes that are deemed to be "Compounds of Concern" for a specific project. "Compounds of Concern" are different from "Target Compounds". "Target Compounds" are all analytes that are

being reported for a site where "Compounds of Concern" are those analytes expected to be present at the site

The number of allowable marginal exceedances is as follows:

1) 11-30 analytes in BS, 1 analyte allowed in ME range;

2) < 11 analytes in BS, no analytes allowed in ME range.

NOTE: SC DHEC does not recognize the concept of Marginal Exceedances. Additionally a secondary check against 70-130% limits should be performed for all analytes reported to SC DHEC.

9.3.2 If the blank spike recoveries are not within the established control limits, the following are required.

9.3.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.3.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.3.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary and the sample results can be reported without qualification.

9.3.2.4 If the recovery of an analyte in the BS is high and the associated sample is non-detect, the data may be reportable. For any DoD QSM projects the resulting data must be qualified accordingly.

9.3.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

9.3.2.6 If there is insufficient sample to re-extract, or if the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.4 Proficiency Test Sample (DoD QSM soil projects)

9.4.1 The proficiency test sample is a bulk volume soil sample. The PT sample is prepared by an outside vendor at specific analyte concentrations. The PT sample

is taken through all preparatory procedures including drying, sieving, grinding, and subsampling.

The surrogate standard is added prior, and the PT sample is then extracted with the other samples to monitor the efficiency of the entire procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = (PT Sample Amount / Amount Spiked) X 100

The percent recovery for each analyte of interest should fall within the Vendors established control limits for the results to be acceptable.

9.4.2 If the PT sample recoveries are not within the established control limits, the following are required.

9.4.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.4.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample.

9.4.2.3 Check to see if the recoveries that are outside of control limits are analytes of concern. If the analytes are not being reported, additional corrective action is not necessary and the sample results can be reported without qualification.

9.4.2.4 If the recovery of an analyte in the PT sample is high and the associated sample is non-detect, the data may be reportable. For any DoD QSM projects the resulting data must be qualified accordingly.

9.4.2.5 If no problem is found, the department supervisor shall review the data and determine what further corrective action is best for each particular sample. That may include reanalyzing the samples, re-extracting and reanalyzing the samples, or qualifying the results as estimated.

9.4.2.6 If the sample is re-extracted beyond hold time, the appropriate footnote and qualifiers should be added to the results. This must be approved by the department supervisor.

9.5 Matrix Spike and Matrix Spike Duplicate

9.5.1 Matrix spike and spike duplicates are replicate sample aliquots to which the surrogate standard and spike standard have been added. The matrix spike and spike duplicate are then extracted and taken through all cleanup procedures along

with the other samples to monitor the precision and accuracy of the extraction procedure. The percent recovery for each analyte is calculated as follows:

% Recovery = [(Spike Amount – Sample Amount) / Amount Spiked] X 100

The percent recovery for each analyte of interest must fall within the established control limits for the results to be acceptable.

9.5.2 If the matrix spike recoveries are not within the established control limits, the following are required.

9.5.2.1 Check to be sure that there are no errors in calculations, dilutions, integrations, or spike solutions. If errors are found, recalculate the data accordingly. If errors are suspected, re-vial and re-inject the extract to verify.

9.5.2.2 Check instrument performance. It may be necessary to re-vial and re-inject the extract in order to verify performance. If an instrument performance problem is identified, correct the problem and reanalyze the sample. If the recovery is high due to interfering peaks, it may be possible to get a more accurate recovery by analyzing the sample on a different column type.

9.5.2.3 If no problem is found, compare the recoveries to those of the blank spike. If the blank spike recoveries indicate that the problem is sample related, document this on the run narrative. Matrix spike recovery failures are not grounds for re-extract but are an indication of the sample matrix effects.

9.5.3 Precision

Matrix spike and spike duplicate recoveries for each analyte are used to calculate the relative percent difference (RPD) for each compound.

RPD = [| MS Result – MSD Result | / Average Result] X 100

The RPD for each analyte should fall within the established control limits. If the RPDs fall outside of the established control limits, the sample, MS and/or MSD should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary.

9.6 Sample Duplicate

The sample duplicate is a replicate sample aliquot to which the surrogate standard has been added. Sample duplicate is then extracted and taken through all cleanup procedures along with the other samples to monitor the precision of the extraction procedure.

Sample and sample duplicate results for each analyte are used to calculate the relative percent difference (RPD) for each compound.

RPD = [| Sample Result – DUP Result | / Average Result] X 100

The RPD for each analyte should fall within the established control limits. If the RPDs fall outside of the established control limits, the sample and/or DUP should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the RPDs are still outside of the control limits, the department supervisor shall review the data and determine if any further action is necessary.

9.7 Sample Triplicate (DoD QSM soil projects)

The sample triplicate is an additional replicate sample aliquot to which the surrogate standard has been added. Sample triplicate is then extracted and taken through all cleanup procedures along with the other samples to monitor the precision of the extraction procedure.

Sample, sample duplicate, and sample triplicate results for each analyte are used to calculate the percent relative standard deviation (%RSD) for each compound.

%RSD = [Standard Deviation of the Result / Average Result] X 100

The %RSD for results above the reporting limit must not be greater than 20%. If the %RSD is greater than 20%, the sample, DUP, and/or TRP should be reanalyzed to ensure that there was no injection problem. If upon reanalysis the %RSD is still greater than 20%, the department supervisor shall review the data and determine if any further action is necessary.

9.8 Grinding Blanks (DoD QSM soil projects)

9.8.1 The grinding blanks (GB) are aliquots of blank sand that are processed through the ring and puck mill between different samples. They are used to monitor for carry over between samples ground with the same bowl set. The grinding blanks for each bowl set may be composited prior to analysis.

9.8.2 The grinding blanks must be free of any analytes of interest or interferences at ½ the required LLOQ to be acceptable. If the grinding blanks are not acceptable, corrective action must be taken to determine the source of the contamination. Samples associated with a contaminated grinding blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the individual grinding blanks (non-composited), reanalyzing the samples or qualifying the results with a “B” or “V” qualifier. This must be approved by the department supervisor.

9.8.3 If the grinding blank is contaminated but the samples are non-detect, then the source of contamination should be investigated and documented. The sample results can be reported without qualification.

10.0 CALCULATIONS

The concentration of each explosive in the original sample is calculated as follows:

Water (ug/l) = $(CONC_{inst})$ X (V_F / V_I) X DF

Soil (ug/kg) = $[(CONC_{inst})$ X (V_F / W_I) X DF]

$CONC_{inst}$	=	Instrument concentration calculated from the initial calibration using mean CF or curve fit
DF	=	Dilution Factor
V_F	=	Volume of final extract (ml)
V_I	=	Volume of sample extracted (ml)
W_I	=	Weight of sample extracted (g)

Soils are air dried prior to extraction; therefore, %solids is not used in the calculation.

11.0 SAFETY AND POLLUTION PREVENTION

11.1 Safety

The analyst should follow normal safety procedures as outlined in the SGS Health and Safety Plan and Personal Protection Policy, which includes the use of safety glasses, gloves, and lab coats.

The toxicity of each reagent and target analyte has not been precisely defined; however, each reagent and sample should be treated as a potential health hazard. Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and many of the target analytes. Exposure must be reduced to the lowest possible level. Personal protective equipment should be used by all analysts.

11.2 Pollution Prevention

Wastewater, methanol, and acetonitrile from the instrument are collected in waste storage bottles and are eventually transferred to the non-chlorinated waste drum.

Sample Extracts are archived and stored for 60 days after analysis. Old extracts and standards are disposed of in the waste vial drum.

12.0 REFERENCES

SW846 Method 8000D Revision 4, July 2014

SW846 Method 8330A Revision 1, February 2007

SW846 Method 3535A Draft Revision 1A, November 1998

SW846 Method 8332 Revision 0, December 1996

SW846 Method 8330B Revision 2, October 2006

DoD Quality System Manual, Version 4.1, April 2009

DoD Quality System Manual, Version 5.0, July 2013

DoD Quality System Manual, Version 5.1, January 2017

METALS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY

Prepared by:	David Metzgar III	Date:	06/21/2016
Approved by:	Svetlana Izosimova	Date:	06/21/2016

Annual Review

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TITLE: METALS BY INDUCTIVELY COUPLED PLASMA ATOMIC EMISSION SPECTROMETRY

REFERENCES: SW846 6010C, EPA 200.7 Rev 4.4 1994

REVISED SECTIONS: Section 2.0: "preserved within 24hrs" changed to "as soon as possible"
Section 1.10: added IDL detail (performed quarterly)

INSTRUMENT: THERMO 6500, SERIAL # 20100903 SSTRACE 1
INSTRUMENT: THERMO 6500, SERIAL # 20103825 SSTRACE 2
AUTOSAMPLER: CETAC 240 POSITION, SERIAL # 031038A520 SSTRACE 1
AUTOSAMPLER: CETAC 240 POSITION, SERIAL # 041048A520 SSTRACE 2

SUGGESTED WAVELENGTH (S): TABLE 2

1.0 SCOPE AND APPLICATION

SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

Method EPA 200.7 has been modified within the flexibility allowed in 40CFR136.6.

1.1 This method is applicable for the determination of metals in water, sludges, sediments, and soils. Elements that can be reported by this method include: Aluminum, Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Titanium, Thallium, Tin, Vanadium, and Zinc.

1.2 Sample matrices are pretreated following SW846 and EPA methods for digestion of soil, sediment, sludge or water samples. Refer to specific metals department digestion SOP's for more information on digestion techniques.

1.3 This inductively coupled argon plasma optical emission spectrometer (s) (ICP-OES) uses an Echelle optical design and a Charge Injection Device (CID) solid-state detector to provide elemental analysis. Control of the spectrometer is provided by PC based iTEVA software. In the instrument, digested samples are introduced into the Thermo 6500 ICP, passed through a nebulizer and transported to a plasma torch. The element-specific emission spectra are produced by a radio frequency inductively coupled plasma. The spectra are dispersed by a spectrometer, and the intensities of the emission lines are monitored with the solid state detector.

1.4 Reporting limits (RL) are based on the extraction procedure. Reporting limits may vary depending on matrix complications, volumes and by client needs, but the reporting limits must always be verified with a low check which meets the criteria outlined in this SOP. Solid matrices are reported on a dry weight basis. Refer to table 1 of this SOP for SGS Accutest - Orlando typical reporting limits. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific reporting limits.

1.5 MDLs must be established for all analytes, using a solution spiked at approximately 3 times the estimated detection limit. To determine the MDL values, take seven replicate aliquots of the spiked sample and process through the entire analytical method. The MDL is calculated by multiplying the standard deviation of the replicate analyses by 3.14, which is the student's t value for a 99% confidence level. MDLs must be determined approximately once per year for each matrix and instrument. Please refer to SGS Accutest - Orlando QA SOP QA020, current version for further information regarding method performance criteria and experimental method detection limits.

1.6 An MDL check standard will be analyzed at the time of the annual MDL study and on a quarterly basis for verification. The concentration of the MDL check standard must be 1x-4x the statistical MDL. The MDL Check Standard is carried through the entire preparation and analytical procedure. This is a qualitative check; therefore, the analyte needs to be detected only. If the analyte is not detected, the concentration of the MDL check standard must be increased to a level where the analyte is detected. This then becomes the current MDL.

1.7 Lower limit of quantitation check sample. The lower limit of quantitation check (LLQC) sample should be analyzed after establishing the lower laboratory reporting limits and on a quarterly basis to demonstrate the desired detection capability. The LLQC sample is carried through the entire preparation and analytical procedure. Lower limits of quantitation are verified when all analytes in the LLQC sample are detected within 20 percent of their true value.

1.8 MDLs are generated for each matrix on both ICP instruments. The higher of the two statistically calculated MDL's is entered into LIMS as the MDL. The verified MDLs are stored in the LIMS and must be at least 2 to 3 times lower than the RL. Exceptions may be made on a case by case basis; however, at no point shall the MDL be higher than the reported RL.

1.9 Compounds detected at concentrations between the RL and MDL are quantitated and qualified as estimated values and reported with either a "J" or "I" qualifier. Some program or project specifications may require that no values below the RL be reported.

1.10 Instrument Detection Limits (IDL). IDL's should be completed upon initial instrument installation and on a quarterly basis. The Instrument Detection Limits (in ug/L) are determined by analyzing 7 replicates of a reagent blank solution on 3 non consecutive days. The IDL is defined as 3 times the average of the standard deviation of the 3 days. Each IDL measurement shall be performed as though it were a separate analytical sample. IDLs shall be determined and reported for each wavelength used in the analysis of the samples.

2.0 PRESERVATION AND BOTTLEWARE

All samples should be preserved with nitric acid to a pH of <2 at the time of collection. All sample pH are checked in sample receiving and within the metals department. Samples that are received with a pH >2 must be preserved to pH <2 and held for 24 hours prior to metals digestion to dissolve any metals that absorb to the container walls. Refer to SOP SAM101, current revision for further instruction. Final pH of TCLP extracts are checked and recorded in SGS Accutest - Orlando Extractions Department. Please refer to TCLP (1311) fluid determination logbook and SPLP (1312) fluid determination logbook for further information. TCLP extracts received from SGS Accutest - Orlando Extractions Department are prepared as soon as possible, no longer than 24 hours from time of receipt. If precipitation is observed during the sample preparation process the sample(s) are immediately re-prepped on dilution until no precipitation is observed. Samples received for dissolved metals analysis should be filtered and preserved to pH<2 as soon as possible and held for 24 hours prior to digestion. Refer to SGS Accutest - Orlando Sample Filtration Logbook for further information.

All soil samples must be stored in a refrigerator at $\leq 6^{o}C$ upon receipt. Refer to SOP SAM101, current revision for further instruction.

All bottleware used by SGS Accutest - Orlando is tested for cleanliness prior to shipping to clients. Analysis results must be less than one half the reporting limit to be acceptable. Refer to SOP SAM104, current revision for further instruction.

3.0 HOLDING TIME AND BATCH SIZE

All samples must be prepared and analyzed within 6 months of the date of collection. Refer to appropriate SGS Accutest - Orlando digestion SOP, current revision for batch size criteria.

4.0 INTERFERENCES

Several types of interferences can cause inaccuracies in trace metals determinations by ICP. These interferences are discussed below.

4.1 Spectral interferences are caused by overlap of a spectral line from another element, unresolved overlap of molecular band spectra, background contribution from continuous or recombination phenomena, and background contribution from stray light from the line emission of high concentration elements. Corrections for these interferences can be made by using interfering element corrections, by choosing an alternate analytical line, and/or by applying background correction points. The locations selected for the measurement of background intensity will be determined by the complexity of the spectrum adjacent to the wavelength peak. The locations used for routine measurement must be free of off-line spectral interference or adequately corrected to reflect the same change in background intensity as occurs at the wavelength peak.

Note: Refer to section 17.0 of this SOP for further instruction regarding interfering element correction factor generation.

4.2 Physical interferences can be caused by changes in sample viscosity or surface tension, by high acid content in a sample, or by high dissolved solids in a sample. These interferences can be reduced by making sample dilutions.

4.3 Matrix interferences in high solid samples can be overcome by using an internal standard. Yttrium/Indium mix is used for the Thermo 6500 ICP. The concentration must be sufficient for optimum precision but not so high as to alter the salt concentration of the matrix. The element intensity is used by the instrument as an internal standard to ratio the analyte intensity signals for both calibration and quantitation.

4.4 Chemical interferences are not pronounced with ICP due to the high temperature of the plasma, however if they are present, they can be reduced by optimizing the analytical conditions (i.e. power level, torch height, etc.).

5.0 APPARATUS

5.1 Currently there are two solid state ICPs available for use in the lab. Both are Thermo 6500 ICP units. These units have been optimized to obtain lower detection limits for a wide range of elements. Since they are solid state systems, different lines may be included for elements to obtain the best analytical results. However, the lines which are normally included in the normal analysis program are shown in Table 2.

5.2 Instrument auto samplers. For random access during sample analysis.

5.3 Class A volumetric glassware and pipettes.

5.4 Polypropylene auto sampler tubes.

5.5 Eppendorf Pipette (s) - Pipette (s) are checked daily for accuracy and to ensure they are in good working condition prior to use. Volumes are checked at 100% of maximum volume (nominal volume). Pipettes are checked within the metals department and results are stored electronically in the “Pipette Calibration Log”. Refer to SOP QA006, current revision for further information regarding pipette calibration. BIAS: mean must be within 2% of nominal volume. Precision: RSD must be $\leq$ 1% of nominal volume based on three replicates.

5.6 Fisher Brand 0.45 micron (um) filter or equivalent. Filter lots are checked for cleanliness through the Method Blank process. All Method Blank analytical results must be less than one half the reporting limit to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated filters must be re-filtered through acceptable filters.

5.7 Fisher Brand disposable 10 ml syringes or equivalent. Syringe lots are checked for cleanliness through the Method Blank process. All Method Blank results must be less than one half the reporting limit to be acceptable, if not , the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated syringes must be re-filtered through acceptable syringes.

5.8 Data System

Microsoft Windows XP Professional Version 2002
Instrument software SST1 – Thermo iTEVA version 2.8.0.89
Instrument software SST2 – Thermo iTEVA version 2.7.0.87

5.8.1 A computer system interfaced to the Thermo 6500 ICP that allows for the continuous acquisition and storage of all data obtained throughout the duration of the analytical run sequence.

5.8.2 Data is archived to a backup server for long term storage.

6.0 REAGENTS

All chemicals listed below are trace metal grade unless otherwise specified. Refer to Acid Certificate of Analysis logbook for Certificates of Analysis and compliance with the specifications of the grade listed. SGS Accutest - Orlando produces DI water to the specifications for the ASTM Type II standard designation based on the system manufacturer's performance specifications. The DI water is used exclusively for laboratory purposes. De-ionized (DI) water should be used whenever water is required. Refer to SOP QA037, current revision for more information regarding testing and monitoring. Refer to the Metals Department Standard Prep Logbook for the make-up and concentrations of standards and stock solutions being used within this SOP. Some of the information included in the logbook is as follows: standard name, elements in mix, manufacturer, lot number, parent expiration date, acid matrix, stock concentration, volume of standard added, total volume, final prepared concentration, prep date, initials, MET number, and prepared standard expiration date. Standards and prepared reagents must be prepared every 6 months or before stock standard expiration date, whichever comes first**.** Refer to tables 3 through 7 of this SOP for concentration levels of standards used. Unless otherwise approved, the calibration curve must contain 3 points determined by a blank and a series of standards representing the elements of interest.

6.1 2.5 ppm Yttrium and 10 ppm Indium internal standard, made from ICP quality standard.

6.2 Hydrochloric acid, trace metals grade.

6.3 Nitric Acid, trace metals grade.

6.4 ICP quality standard stock solutions are available from Inorganic Ventures, Spex, Plasma Pure, Ultra, Environmental Express, or equivalent.

6.5 Calibration Standards. These can be made up by diluting the stock solutions to the appropriate concentrations. The calibration standards should be prepared using the same type of acid (s) and at approximately the same concentration as will result in the samples following sample preparation.

6.5.1 For calibration and quantitation an internal standard (Yttrium/Indium) is used to limit nebulization problems. If it is known that the samples contain a significantly different

acid matrix, the samples must be diluted so that they are in a similar matrix to the curve. All sample results are referenced to the initial calibration blank (ICB) Internal Standard counts. The criteria is 60-125 percent of the initial calibration blank (ICB) counts. If the internal standard counts fall outside these criteria matrix effects must be suspected and the sample diluted until it meets the criteria or footnoted in LIMS as suspected matrix interference.

6.5.2 Standards must be prepared so that there is minimal spectral interference between analytes.

Note: All Ag stock and intermediate solutions must be stored away from direct sunlight.

6.6 Analytical Quality Control Solutions.

All of the solutions below are prepared by adding either mixed or single element metals solutions to a solution prepared using the same type of acid (s) and at approximately the same concentration as will result in the samples following sample preparation.

6.6.1 Blank (Calibration, ICB, CCB)

This reagent blank contains Nitric Acid at 3 percent and Hydrochloric Acid at 5 percent.

6.6.2 Initial Calibration Verification solution.

This standard solution must be made from a different source than the calibration curve. The concentrations for each element must be within the range of the calibration curve and should be approximately at the midpoint of the curve. This solution is used to verify the accuracy of the initial calibration. Levels for the ICV standard are shown in Table 4.

6.6.3 Continuing Calibration Verification solution.

The metals concentrations for this standard should be at approximately the mid point of the calibration curve for each element. This standard should be prepared from the same source that is used for the calibration curve. Levels for the CCV standard are shown in Table 5.

6.6.4 Interference Element Check Solutions.

These solutions must be analyzed to check the interfering element correction factors (IEC's) on the ICP instruments. Refer to section 17.0 of this SOP for further information regarding generation of IEC's.

6.6.4.1 ICSA Solution.

The ICSA solution contains only the interfering elements. Levels for the ICSA are shown in Table 9.

6.6.4.2 ICSAB Solution.

The ICSAB solution contains both the interferents and the analytes of interest. Levels for the ICSAB are shown in Table 10.

6.6.4.3 Single element interference check solutions

Prepared as single solutions. Levels for the single element interference solutions are shown in Table 11.

6.7 CRIA Standard Solution (Also referred to as LLCCV)

The CRIA standard contains the elements of interest at levels equal to SGS Accutest - Orlando quantitation limits (RL). Please refer to Table 6 for list of elements of interest and concentration levels for the CRIA. If special client reporting limits are requested, then low checks corresponding to those reporting limits must also be analyzed.

6.8 Matrix Spike, Matrix Spike duplicate, and Spike Blank Solution.

This solution is prepared by adding either mixed or single element metals solutions to a solution containing 3 percent nitric acid and 5 percent hydrochloric acid and diluting to a fixed final volume with this acid mixture. Spiking solution (s) must be added to the spike blank, matrix spike, and the matrix spike duplicate prior to digestion. Levels for the MS and MSD and Spike Blank standard are shown in Table 7.

6.9 Liquid Argon or Argon Gas. (99.999% purity)

7.0 ANALYTICAL PROCEDURE

Note: Please refer to section 8 of this SOP for further detail on quality control standards. Please refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements.

7.1 General procedure on how to operate the Thermo 6500 is described below. Refer to the Thermo 6500 operation manual for further details.

7.2 Before starting up the instrument, make sure that the pump tubing is in good condition, the torch assembly, the nebulizer, and the spray chamber are clean, the dehumidifier (if used) is filled with DI water up to the level between Minimum and Maximum, and that there are no leaks in the torch area.

7.3 Turn on the recirculating cooler. Verify that the argon is turned on and there is enough for the entire days analytical run.

7.4 Tighten the pump platens and engage the peristaltic pump. Make sure sample and internal standard solutions are flowing smoothly.

7.5 Put a new solution of acid rinse into the rinse reservoir. The composition of the rinse solution may be periodically changed to minimize sample introduction problems and sample carryover. If internal standard is being used, make sure that sufficient amount of internal standard is prepared for the entire analytical run.

7.6 Start up the instrument following the sequence show below.

7.6.1 Double click the **iTEVA Control Center** Icon on the desktop. Type ***admin*** in User Name field, and then click **OK**.

7.6.2 Once the iTEVA Control Center window is opened, click on **Plasma** Icon at status bar area. Then click on **Instrument Status** to check the interlock indicators (torch compartment, purge gas supply, plasma gas supply, water flow and exhaust should be in green; drain flow and busy should be in gray) and the Optics Temperature. (It should be around 38°C.) Click on the Close box.

7.6.3 Click **Plasma On**. When the plasma is on, click close. Let the instrument warm up for 15 to 20 minutes before starting the analysis. New tubing may take an hour to stabilize.

7.7 Torch Alignment and Auto Peak

7.7.1 If the torch has been cleaned, then the torch alignment procedure must be performed.

7.7.2 Open the method and then click on **Sequence** tab, then click on **List View** Icon until you reach rack display.

7.7.3 Go to S-6 position (you can assign any position in the rack for torch alignment), then right click to select **Go** to empty sample S:6. (Now, the auto sampler tip moves from Rinse to this position).

7.7.4 Click on **Analysis** tab, then select **Torch Alignment** from Instrument drop down menu. There will be a pop up dialog box present. Click **Run**. Then there will be another dialog pop up box (This is a reminder for Torch Alignment Solution (2 ppm Zn)), click **Ok**. Now, the instrument is initializing an automated torch alignment. It takes about 7 minutes to complete this step. Progress is indicated in the progress bar.

7.7.5 After torch alignment is complete, click **Close**. Click on **Sequence** tab, then followed by **List View** Icon.

7.7.6 Go to Rinse position at rack display, right click to select Go to rinse and let it rinse for approximately 5 minutes.

7.7.7 Perform Auto Peak

7.7.8 It is recommended that the Auto Peak Adjust procedure be performed daily prior to calibration. A standard that contains all of the lines of interest is used and the system automatically makes the appropriate fine adjustments. (High standard solution should be used for this process.)

7.7.9 Click **Sequence** tab, then click on **List View** Icon until the rack is displayed.

7.7.10 Go to S-5 position (you can assign any position in the rack for auto peak adjust), then right click to select **Go** to empty sample S:5. (Now, the auto sampler tip moves from the Rinse position to this position). Click on **Analysis** tab. All elements result is shown in the display area. From Instrument drop down menu, select **Perform Auto Peak**. There will be a pop up dialog box present. Highlight "All Elements", and then click **Run**. Then there will another pop up dialog box (This is a reminder for Auto Peak Solution), click **Ok**. Now, the instrument is performing auto peak adjust. It takes about 5 minutes to complete this process. The Auto Peak dialog box will show a green check mark in front of "All Elements", which indicates Auto Peak is complete.

7.8 Open the method and start up the run.

7.8.1 Click on **Analyst** Icon at the workspace. Go to the method and choose Open from the drop down menu. Select the method with the latest revision number.

7.8.2 Go to **Method** tab at the bottom of left hand corner to click on **Automated Output** at the workspace area. Type a filename in Filename field in the data display area (i.e. : SA101010M1, starts with SA, then followed by MM-DD-YY, then M1; M1 indicates the first analytical run for that day, then followed by M2, M3 and so on for the second and third runs.) Click on **Apply To All Sample Types**.

7.8.3 Click on **Sequence** tab at the bottom of left hand corner. From Auto Session drop down menu bar, click on **New Auto sampler** to create a sequence. This will pop up a dialog box, then click on **New** and fill in number of samples (i.e.: 100) in the Number of Samples field and the sample I.D. (leave this field empty) in Sample Name field. Type a sequence name (i.e. : SEQ101010M1, starts with SEQ, then MM-DD-YY, then M1; M1 indicates the first analytical run for that day, then followed by M2, M3 and so on for the second and third runs) in the Sequence Name field. Click Ok, then put in "0" as settle time between between sequences, and click **Ok**.

7.8.4 Right click on **Untitled** (Cetac ASX-520 Enviro 5 Named Rack is the rack that is currently used) at the workspace area, click on **Auto-Locate All** to locate all sample positions.

7.8.5 Double click on **Untitled** again, then click on the sequence name (i.e. : SEQ101010M1), on the data display area, type the sequence in Samplename column, dilution factor (if needed) in CorrFact column, check the box in front of Check column, and select an appropriate check table.

7.8.6 Once done with creating sequence, go to **Method** drop down menu and save all changes as **Save As**. There will be a Save a Method dialog box present, go to the

save option to check on "Overwrite Method and bump revision number" box, and then click **Ok**.

7.8.7 Go to Sequence tab, click on List View Icon from tool bar, then click on Connect Autosampler to PC and Initialize Icon.

7.8.8 See table 8 for a typical run sequence.

7.9 Calibrate the instrument as outlined below. See table 3 for calibration standards concentrations. This calibration procedure is done a minimum of once every 24 hours. The calibration standards may be included in the auto sampler program or they may be run manually from the **Calibrate Instrument (graduated cylinder)** icon located on the Analyst tab. All curves must be determined from a linear calibration prepared in the normal manner using the established analytical procedure for the instrument. Refer to instrument manual for further detail. Unless otherwise approved, the calibration curve must be determined by a blank and a series of three standards representing the elements of interest. Three exposures will be used with a percent relative standard deviation of less than 5 percent. The resulting correlation coefficient must be $\geq$0.998. If the calibration curves do not meet these criteria, analysis must be terminated, the problem corrected, and instrument re-calibrated. Correlation coefficients, slopes, and y-intercepts for each wavelength are printed and included in each analytical data package.

7.10 After the instrument is properly calibrated, begin by reanalyzing the high standard(s) for each element. The standards can be combined into one solution for this analysis. The analyzed value must be within 5 percent of the true value or that element must be re-calibrated. The High Standard Check shall be used for 200.7 only. After the high standards are analyzed, the ICV check standard shall be run. For the ICV, all elements to be reported must be within 5 percent of the true value for 200.7 and 10 percent of the true value for 6010C. If the ICV fails, analysis shall be terminated, problem corrected, and the instrument re-calibrated.

7.11 After analyzing the ICV, the ICB must be analyzed. The results of the ICB must be less than one half the reporting limit. The instrument blank may be failing the criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank, qualifying the results with a "B" or "V" qualifier, or raising the reporting limit for all samples to greater than two times the background concentration.

7.12 Before analyzing any real samples the CRIA (also referred to as LLCCV) must be analyzed. The CRIA contains elements of interest at the reporting limit. The CRIA will be analyzed at the beginning and end of each analytical run. For all elements the results must be within 20 percent of the true value for client specific reporting limits (CRIA Requirement). For all others a 30 percent criterion will be applied. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific reporting limits (CRIA Requirement). If the initial CRIA fails no samples associated with the failing CRIA can be reported, and the CRIA should be reanalyzed for the failing elements. If the closing CRIA fails the criteria, the samples associated with the CRIA shall be evaluated as to the best corrective action for

each particular sample. This may include reanalyzing the samples associated with the CRIA, or qualifying the results in LIMS.

7.13 Before analyzing any real samples, the interference check standards (ICSA, ICSAB) must be analyzed. For all spiked elements, the analyzed results must be within 20 percent of the true value. For non-spiked elements, the interfering element solutions must be ± the absolute value of the reporting limit for each element. Also, on an as needed basis (i.e.: instrument repair), analyze the single element interference check solutions (SIC). The same criteria as outlined above apply. If the ICSA and/or the ICSAB fall outside this criterion the problem must be corrected and the instrument re-calibrated or data footnoted in LIMS system. If the closing ICSA/ICSAB fails the criteria, the samples associated with the ICSA/ICSAB shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples associated with the ICSA/ICSAB, or qualifying the results in LIMS. Refer to section 17.0 of this SOP for Interfering Element Correction (IEC) procedure.

7.14 After the initial analytical quality control has been analyzed, the samples and the preparation batch matrix quality control shall be analyzed. Each sample analysis must be a minimum of 3 readings using at least a 5 second integration time. Between each sample, flush the nebulizer and the solution uptake system with a blank rinse solution for at least 60 seconds or for the required period of time to ensure that analyte memory effects are not occurring.

7.15 Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth sample and at the end of the sample run. If the CCV solution is not within 10 percent of the true value for method 6010C and 5 percent for method 200.7 (for the initial CCV (ICCV)), the CCV shall be reanalyzed to confirm the initial value. If the CCV is not within criteria after the reanalysis, no samples can be reported in the area bracketed by the failing CCV. Immediately following the analysis of the CCV the CCB shall be analyzed. The results of the CCB must be less than one half the reporting limit for all elements. The instrument blank may be failing the criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank, qualifying the results with a “B” or “V” qualifier, or raising the reporting limit for all samples to greater than two times the background concentration.

7.16 One sample per preparation batch, or whenever matrix interferences are suspected for a batch of samples, a serial dilution (SDL) must be prepared. For the serial dilution, a 1:5 dilution must be made on the sample. The results of the 1:5 dilution shall agree within 10 percent of the true value as long as the sample and the dilution result are greater than 10 times the method detection limit or greater than 50 times the IDL. If the results are outside these criteria then matrix interference should be suspected and the proper footnote entered into LIMS. A post digestion spike (PDS) must be performed if the SDL fails. The PDS must recover within ± 20 percent for method SW846-6010C and ± 15 percent for method EPA 200.7. If the PDS is outside these limits then matrix interference must be suspected and the proper footnote entered into LIMS.

7.17 The upper limit of quantitation may exceed the highest concentration calibration point and can be defined as the “linear dynamic” range. Sample results above the linear dynamic range shall be diluted under the linear dynamic range and reanalyzed. Samples following a sample with high concentrations of analyte (s) must be examined for possible carryover.

Verification may be done by rinsing the lines with an acid solution and then reanalyzing the sample. A limit check table is built into the autosampler file so that samples exceeding the standardization range are flagged on the raw data.

7.18 After the instrument is optimized and all initial QC has been run, click on **Run Auto-Session** Icon to start the analytical run sequence.

7.18.1 If you need to add or delete samples once the run is started, follow the steps shown below.

7.18.2 Click on **Sequence** tab, then click on **List View** Icon at the tool bar. There is the sequence table shown on the display area.

7.18.3 Click on **Add Samples** Icon. This will pop up a dialog box, and then fill in number of samples that need to be added. Click **Ok**. By doing this, samples will be added to the end of the current sequence without a rack location.

7.18.4 On the Samplename column type in the sample I.D., correction factors, and check tables. **Click on Auto Locate All.**

7.18.5 The added samples will be analyzed at the end of the original sequence run order unless they are assigned a different run order.

7.18.6 Deleting Samples

7.18.7 Click on **Sequence** tab, and then click on **List View** Icon under the sequence display area.

7.18.8 Highlight all samples that need to be deleted and then click on the **Delete Samples** icon.

7.19 When the analysis is completed export the data to LIMS following the procedure outlined below.

7.19.1 Double click on **ePrint** Icon on desktop. There will be a **LEADTOOLS ePRINT** pop up box, click on **Finish Jobs** and **OK** boxes.

7.19.2 Double click the **PDF** Icon on the desktop; the PDF file will be present as Document_#. Right click on that file, select **rename** to change the filename to an assigned analytical run I.D. (i.e.: MA9000). This is the raw data file for MA9000.

7.19.3 Drop the raw data to the **LIMS Data Drop** icon located on the desktop.

7.19.4 By completing the above steps, the raw data (i.e.: MA9000) can be viewed and/or printed from the Raw Data Search function.

7.19.5 Go to **Analysis** tab, right click on sample header, and select export all samples. A pop up dialog box will come up, type in the analytical run I.D. (i.e.: SA101010M1) and click **Ok**. Go to **LIMS Export** folder located on the desktop, right click on analytical

run and change extension from .TXT to .ICP. Open the analytical file and make any necessary changes, such as deleting any samples that need to be re-run on dilution. **Save** the file. Drop the data file to the **LIMS Data Drop** icon located on the desktop. This will then send the export file to LIMS for review.

7.20 The data can be evaluated by running an automated data evaluation program, which will help to generate quality control summary pages. Each run must be evaluated as quickly as possible to make sure that all required quality control has been analyzed. With each data package include: cover sheet, copies of all prep sheets, autosampler run sequence, dilution sheets, and raw data. Label each folder with MA#, instrument run I.D., instrument used, and date.

7.21 At the end of the analysis day the ICP must be shutdown using the following sequence.

7.21.1 Place the auto sampler tip in the rinse cup and rinse in a mixed solution of approximately 5 percent nitric acid and 5 percent hydrochloric acid for 10 minutes and then in DI water for 20 minutes.

7.21.2 Turn off the plasma by clicking on the **Plasma** Icon and then by clicking **Plasma Off.**

7.21.3 Close all iTeva programs/windows.

7.21.4 Release the tension on the sample pump platens.

7.21.5 Turn off recirculating chiller.

8.0 QUALITY CONTROL

This section outlines the QA/QC operations necessary to satisfy the analytical requirements for method SW846 6010C. Please refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements. Check with the area supervisor or lab manager for any non compliant quality control for further information.

8.1 High Standard Check.

After the instrument is properly calibrated, the high standard(s) shall be reanalyzed for each element. The analyzed value must be within 5 percent of the true value. If the High Standard falls outside this criteria analysis shall be terminated, problem corrected, and the instrument re-calibrated.

Note: High Standard Check is for method 200.7 only. The standards can be combined into one solution for this analysis.

8.2 Initial Calibration Verification Standard (ICV).

After each calibration, a standard from a different source than the calibration standard shall be analyzed. For the ICV, all elements to be reported must be within 10 percent of the true value for 6010C and within 5 percent for 200.7. If the ICV is outside these criteria then the analysis must be terminated, problem corrected, and the instrument re-calibrated.

8.3 Continuing Calibration Blank/Initial Calibration Blank.

Analyze the Initial calibration blank solution at the beginning of each run and the continuing calibration blank after every tenth sample and at the end of the sample run. The ICB/CCB must be less than one half the reporting limit for each element. The instrument blank may be failing the criteria due to contamination or instrument drift. Samples associated with the failing blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples bracketed by the failing blank, qualifying the results with a "B" or "V" qualifier, or raising the reporting limit for all samples to greater than two times the background concentration.

8.4 Low Standard Check (CRIA).

The CRIA (also referred to as LLCCV) contains elements of interest at the reporting limit. The CRIA will be analyzed at the beginning and end of each analytical run. For all elements the results must be within 20 percent of the true value for client specific reporting limits (CRIA Requirement). For all others a 30 percent criterion will be applied. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific reporting limits (CRIA Requirement). If the initial CRIA fails no samples associated with the failing CRIA can be reported, and the CRIA should be reanalyzed for the failing elements. If the closing CRIA fails the criteria, the samples associated with the CRIA shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples associated with the CRIA, or qualifying the results in LIMS.

8.5 ICSA and ICSAB and Single Element Interference Solutions

Analyze the ICSA and ICSAB at the beginning and end of each run following the analysis of the CRIA. Also, on an as needed basis (i.e.: instrument repair), analyze the single element interference check solutions (SIC). For all spiked elements, the analyzed results must be within 20 percent of the true value. For non-spiked elements, the interfering element solutions must be $\pm$ the absolute value of the reporting limit for each element. If the ICSA and/or the ICSAB fall outside this criterion the problem must be corrected and the instrument re-calibrated or data footnoted in LIMS system. If the closing ICSA/ICSAB fails the criteria, the samples associated with the ICSA/ICSAB shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples associated with the ICSA/ICSAB, or qualifying the results in LIMS. Refer to section 17.0 of this SOP for Interfering Element Correction (IEC) procedure.

8.6 Continuing Calibration Verification.

Analyze the continuing calibration verification solution and the continuing calibration blank after every tenth sample and at the end of the sample run. If the CCV solution is not within 10 percent of the true value for method 6010C and 5 percent for method 200.7 (for the initial CCV (ICCV)) the CCV must be reanalyzed to confirm the initial value. If the CCV is not within criteria after reanalysis no samples can be reported in the area bracketed by the failing CCV.

8.7 Method Blank.

The laboratory must digest and analyze a method blank with each batch of samples. The method blank must contain elements at less than one half the reporting limit for each element. The exception to this rule is when the samples to be reported contain greater than 10 times the method blank level. In addition, if all the samples are less than a client required limit and the method blank is also less than that limit, then the results can be reported as less than that limit. Samples associated with the contaminated blank shall be evaluated as to the best corrective action for each particular sample. This may include reanalyzing the samples, re-digesting and reanalyzing the samples, qualifying the results with a "B" or "V" qualifier, or raising the reporting limit to greater than two times the background concentration,

8.8 Blank Spike Sample.

The laboratory must digest and analyze a spike blank sample with each batch of samples. Blank Spikes must be within 20 percent of the true value for method SW846-6010C and within 15 percent for method EPA 200.7. If the lab control is outside of the control limits for a reportable element, all samples must be re-digested and reanalyzed for that element. The exception is if the lab control recovery is high and the results of the samples to be reported are less than the reporting limit. In that case, the sample results may be reported with no flag. For solid standard reference materials (SRMs) $\pm$ 20 percent accuracy may not be achievable and the manufacturer's established acceptance criterion should be used for all soil SRMs.

8.9 Matrix Spike and Matrix Spike Duplicate Recovery.

The laboratory must digest and analyze a matrix spike and matrix spike duplicate with each batch of samples. The matrix spike recovery is calculated as shown below and must be within 20 percent of the true value for method SW846-6010C and within 30 percent for method EPA 200.7. If a matrix spike is out of control, then the results must be flagged with the appropriate footnote. If the matrix spike amount is less than one fourth of the sample amount, then the sample cannot be assessed against the control limits and must be footnoted to that effect.

Note: Both the matrix spike amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

$$\frac{\text{(Spiked Sample Result - Sample Result)}}{\text{Amount Spiked}} \times 100 = \text{matrix spike recovery}$$

8.10 Matrix Duplicate/Matrix Spike Duplicate Relative Percent Difference.

The laboratory must digest a duplicate with each batch of samples. The relative percent difference (RPD) between the duplicate and the sample must be assessed and must be $\leq$ 20 percent for sample results at or above the reporting limit. If the RPD is outside the 20 percent criteria the results must be qualified in LIMS. RPD's are also calculated in LIMS for sample results below the reporting limit. RPD's outside the 20 percent criteria are not considered failing and LIMS automatically footnotes these as "RPD acceptable due to low duplicate and sample concentrations."

Note: Both the duplicate amount and the sample amount are calculated to the IDL for any given element. Any value less than the IDL is treated as zero.

$$\frac{(|\text{Sample Result} - \text{Duplicate Result}|) \times 100}{(\text{Sample Result} + \text{Duplicate Result})/2} = \text{Duplicate RPD}$$

8.11 Serial Dilution Analysis and Post Digestion Spike.

For one sample per preparation batch, or whenever matrix interferences are suspected for a batch of samples, a serial dilution must be prepared. For the serial dilution, a 1:5 dilution must be made on the sample. The results of the 1:5 dilution must agree within 10 percent of the true value as long as the sample and the dilution result are greater than 10 times the method detection limit and/or greater than 50 times the IDL. If the dilution does not agree, then the sample must be post digestion spiked (PDS) at a level no less than 10 times but no greater than 100 times the MDL concentration. The PDS must recover within ± 20 percent for method SW846-6010C and ± 15 percent for method EPA 200.7. If the PDS is outside these limits then matrix interference must be suspected and the proper footnote entered into LIMS.

$$\frac{\text{(Sample Result - Serial Dil. Result)} \times 100}{\text{Sample Result}} = \text{Serial Dilution RPD}$$

8.12 Linear Calibration ranges.

The upper limit of the linear calibration ranges must be established for all elements by determining the signal responses from a minimum of three concentration standards, one of which is close to the upper limit of the linear range. The linear calibration range, which may be used for the analysis of samples must be judged by the analyst from the resulting data. The upper range limit should be an observed signal no more than 10% below the level extrapolated from lower standards. Linear calibration ranges must be determined whenever there is a significant change in instrument response or at a minimum, every 6 months.

8.13 Sample RSD

For samples containing levels of elements greater than five times the reporting limits, the relative standard deviation for the replicates should be less than 5%. If not, reanalyze the sample. If upon reanalysis, the RSD's are acceptable then report the data from the reanalysis. If RSD's are not acceptable upon reanalysis, then the results for that element should be footnoted that there are possible analytical problems and/or matrix interference indicated by a high RSD between replicates.

8.14 Interelement Spectral Interference Correction Validity

For the interelement spectral interference corrections to remain valid during sample analysis, the interferent concentration must not exceed its linear range. If the interferent concentration exceeds its linear range or its correction factor is big enough to affect the element of interest even at lower concentrations, sample dilution with reagent blank and

reanalysis is required. In these circumstances, analyte dilution limits are raised by an amount equivalent to the dilution factor.

8.15 Internal Standard (Yttrium/Indium)

For any readings where the internal standard is outside of the range 60-125 percent of the internal standard level in the reference standard (Initial Calibration Blank), then the sample must be diluted until the internal standard is within range and all sample results must be footnoted in LIMS.

8.16 MSA (Method of Standard Additions)

SGS Accutest - Orlando uses the internal standard technique as an alternative to the MSA per SW846-6010C section 4.4.2. However, in certain circumstances MSA may be needed by some project specific requirements. SGS Accutest - Orlando may perform an MSA when sample matrix interference is confirmed through the post digestion spike process or may qualify the results in LIMS. SGS Accutest - Orlando will use a single addition method as described in SW846-7000B.

9.0 GLASSWARE CLEANING

All glassware must be washed with soap and tap water and then rinsed with 5 percent nitric acid. It must then be rinsed at least 3 times with DI water. Refer to SOP GN196, current revision for further information regarding glassware cleaning.

10.0 DOCUMENTATION REQUIREMENTS

Refer to the Laboratory Quality Assurance Manual for documentation requirements. All raw data is printed to .PDF format and archived to a backup server for long term storage.

11.0 SAFETY

The analyst must follow normal safety procedures as outlined in the SGS Accutest - Orlando Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and must be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact the supervisor. Follow proper safety precautions when working with gas cylinders.

12.0 CALCULATIONS

For water samples, the following calculations must be used. Refer to the QC section for the calculations to be used for the QC samples.

Original sample concentration of metal (ug/l) =

$$\frac{\text{(conc. in the digestate (ug/l)) x (final digestate volume (ml))}}{\text{(initial sample volume (ml))}}$$

For soil samples, the following calculations must be used.

Concentration of the metal in the dry sample (mg/kg) =

$$\frac{\text{(conc. in the digestate (mg/l) x final digestate volume(L))}}{\text{(sample wt. (kg)) x (\% solids/100)}}$$

13.0 INSTRUMENT MAINTENANCE

Recommended periodic maintenance includes the items outlined below. All maintenance must be recorded in the instrument maintenance log.

13.1 Change the pump tubing as needed.

13.2 Clean the filter on the recirculating pump approximately once a month and dust off the power supply vents as needed.

13.3 Clean or replace the nebulizer, torch assembly, and injector tube as needed.

13.4 Change the sampler tip as needed.

13.5 Clean the recirculating pump lines and internal sock filter every 3 months or as needed.

13.6 Clean the radial view quartz surface weekly or more often if needed.

14.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

14.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids must be followed. All method users must be familiar with the waste management practices described in Section 14.2.

14.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

15.0 GENERIC DEFINITIONS

15.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 24 hours whichever comes first.

15.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

15.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. A CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

15.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

15.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point must be at a level equal to or below the reporting level.

15.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor must be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

15.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the performance of a method in a given sample matrix.

15.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and performance of a method in a given sample matrix.

15.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

15.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

15.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

16.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Statistical control limits are stored in the LIMS for QA purposes only. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

17.0 GENERATION OF INTERFERING ELEMENT CORRECTION FACTORS

17.1 It is recommended that all IEC's be verified and updated approximately every 6 months or whenever instrument conditions change significantly. It is also recommended that elements with frequent high concentrations or with large IEC's should be checked more frequently.

17.2 Calculate the IEC correction factors and enter them into the method (refer to Thermo 6500 instrument manual). Calculate the correction factor using the equation shown below. This correction factor must be added to the correction factor already in place in the method for a given element.

$$\text{IEC} = \frac{\text{Concentration Result of the element with the interference}}{\text{Concentration result of the interfering element}}$$

17.3 Verify the new correction factors by reanalyzing the ICSA/ICSAB solutions and/or the SIC solutions or by reloading and recalculating the previously stored results. If the reanalysis is not within QC limits, make additional changes to the IEC factors and then re-verify both the individual and combined solution values.

17.4 Save and update the method.

17.5 Interfering element correction factors are saved as raw data along with the run printouts on a daily basis so that the IEC's for a given run are traceable.

TABLE 1: REPORTING LIMIT BY ELEMENT

Analyte	Water Reporting Limit (ug/L)	Soil Reporting Limit (mg/kg)	TCLP Reporting Limit (mg/L)/MCL
Tin	50	5	
Aluminum	200	20	
Antimony	5	1	
Arsenic	10	0.5	0.10 / 5.0
Barium	200	20	10 / 100
Beryllium	4	0.5	
Cadmium	5	0.4	0.05 / 1.0
Calcium	1000	500	
Chromium	10	1	0.10 / 5.0
Cobalt	50	5	
Copper	25	2.5	
Iron	300	10	
Lead	5	1	0.5 / 5.0
Magnesium	5000	500	
Manganese	15	1.5	
Nickel	40	4.0	
Potassium	5000	500	
Selenium	10	1	0.5 / 1.0
Silver	10	1	0.10 / 5.0
Sodium	5000	500	
Thallium	10	1	
Vanadium	50	5	
Zinc	20	2	
Molybdenum	50	2.5	
Strontium	10	0.5	
Titanium	10	0.5	

TABLE 2. THERMO 6500 ANALYSIS LINES

Element	Wavelength
Al	396.1
As	189.042
Ca	317.933
Fe	259.9
Mg	279.078
Mn	257.610
Pb	220.353
Se	196.026
Tl	190.864
V	292.402
Ag	328.068
Ba	455.4
Be	313.042
Cd	226.502
Co	228.616
Cr	267.716
Cu	324.753
K	766.491
Na	589.5
Ni	231.604
Sb	206.838
Zn	206.2
Mo	202.030
Sn	189.900
Sr	407.7
Ti	334.9

TABLE 3: LOW, MID AND HIGH STANDARD LEVELS

Element	Low	Mid	High
	ug/l	ug/l	ug/l
Al	10000	40000	80000
As	500	2000	4000
Ca	10000	40000	80000
Fe	10000	40000	80000
Mg	10000	40000	80000
Mn	500	2000	4000
Pb	500	2000	4000
Se	500	2000	4000
Tl	500	2000	4000
V	500	2000	4000
Ag	62.5	250	500
Ba	500	2000	4000
Be	500	2000	4000
Cd	500	2000	4000
Co	500	2000	4000
Cr	500	2000	4000
Cu	500	2000	4000
K	10000	40000	80000
Na	10000	40000	80000
Ni	500	2000	4000
Sb	500	2000	4000
Zn	500	2000	4000
Mo	500	2000	4000
Sn	500	2000	4000
Sr	500	2000	4000
Ti	500	2000	4000

TABLE 4: ICV STANDARD LEVELS

Element	Concentration
	ug/l
Al	40000
As	2000
Ca	40000
Fe	40000
Mg	40000
Mn	2000
Pb	2000
Se	2000
Tl	2000
V	2000
Ag	250
Ba	2000
Be	2000
Cd	2000
Co	2000
Cr	2000
Cu	2000
K	40000
Na	40000
Ni	2000
Sb	2000
Zn	2000
Mo	2000
Sn	2000
Sr	2000
Ti	2000

TABLE 5: CCV STANDARD LEVELS

Element	Concentration
	ug/l
Al	40000
As	2000
Ca	40000
Fe	40000
Mg	40000
Mn	2000
Pb	2000
Se	2000
Tl	2000
V	2000
Ag	250
Ba	2000
Be	2000
Cd	2000
Co	2000
Cr	2000
Cu	2000
K	40000
Na	40000
Ni	2000
Sb	2000
Zn	2000
Mo	2000
Sn	2000
Sr	2000
Ti	2000

TABLE 6: CRIA STANDARD LEVELS

Element	CRIA
	ug/l
Al	200
As	10
Ca	1000
Fe	300
Mg	5000
Mn	15
Pb	5
Se	5
Tl	10
V	50
Ag	10
Ba	200
Be	5
Cd	5
Co	50
Cr	10
Cu	25
K	5000
Na	5000
Ni	40
Sb	5
Zn	20
Mo	50
Sn	50
Sr	10
Ti	10

TABLE 7: BLANK SPIKE, MATRIX SPIKE AND MATRIX SPIKE DUPLICATE LEVELS

Element	Concentration
	ug/l
Al	27000
As	2000
Ca	25000
Fe	26000
Mg	25000
Mn	500
Pb	500
Se	2000
Tl	2000
V	500
Ag	50
Ba	2000
Be	50
Cd	50
Co	500
Cr	200
Cu	250
K	25000
Na	25000
Ni	500
Sb	500
Zn	500
Mo	500
Sn	500
Sr	500
Ti	500

TABLE 8: TYPICAL RUN SEQUENCE

BLANK
LOW
MID
HIGH
HIGH STD
ICV
ICB
CRIA
ICSA
ICSAB
CCV
CCB
MB
SB
SAMPLE1
DUPLICATE
SERIAL DILUTION
MATRIX SPIKE
MATRIX SPIKE DUPLICATE
POST DIGESTION SPIKE
SAMPLE2
SAMPLE3
CCV
CCB
SAMPLE4
SAMPLE5
SAMPLE6
SAMPLE7
SAMPLE8
SAMPLE9
SAMPLE10
SAMPLE11
SAMPLE12
SAMPLE13
CRIA CLOSING
ICSA CLOSING
ICSAB CLOSING
CCV
CCB

TABLE 9: ICSA SOLUTION LEVELS

Element	Concentration
	mg/l
Al	500
As	0
Ca	500
Fe	200
Mg	500
Mn	0
Pb	0
Se	0
Tl	0
V	0
Ag	0
Ba	0
Be	0
Cd	0
Co	0
Cr	0
Cu	0
K	0
Na	0
Ni	0
Sb	0
Zn	0
Mo	0
Sn	0
Sr	0
Ti	0

TABLE 10: ICSAB SOLUTION LEVELS

Element	Concentration
	mg/l
Al	500
As	1.0
Ca	500
Fe	200
Mg	500
Mn	0.5
Pb	1.0
Se	1.0
Tl	1.0
V	0.5
Ag	1.0
Ba	0.5
Be	0.5
Cd	1.0
Co	0.5
Cr	0.5
Cu	0.5
K	0
Na	0
Ni	1.0
Sb	1.0
Zn	1.0
Mo	1.0
Sn	1.0
Sr	1.0
Ti	1.0

TABLE 11: SINGLE ELEMENT INTERFERENCE CHECK SOLUTION (SIC) LEVELS

Element	Concentration
	mg/l
Al	500
As	0
Ca	500
Fe	200
Mg	500
Mn	0
Pb	0
Se	0
Tl	0
V	0
Ag	0
Ba	0
Be	0
Cd	0
Co	0
Cr	0
Cu	0
K	0
Na	0
Ni	0
Sb	0
Zn	0
Mo	0
Sn	0
Si	50
Sr	0
Ti	0

DIGESTION OF WATER SAMPLES FOR ICP ANALYSIS

Prepared by: (b) (6) Date: 06/21/2016

Approved by: Date: 06/21/2016

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TITLE: DIGESTION OF WATER SAMPLES FOR ICP ANALYSIS

REFERENCES: SW846 3010A, EPA 200.7, EPA 200.8

REVISED SECTIONS: SOP rebranded

1.0 SCOPE AND APPLICATION, SUMMARY

This method is applicable for the digestion of aqueous samples, TCLP extracts and wastes that contain small amounts of suspended solids. After digestion, the samples can be analyzed by ICP. The digestion methods described in this SOP are based upon SW846 method 3010A, EPA 200.7 March 1983, and EPA 200.8, revision 5.4, 1994 digestion methods.

Reduced volume versions of methods 200.7, March 1983 and method 200.8, Rev.5.4 1994 are in use by ASE. This approach that uses the same reagents and molar ratios is acceptable by the regulatory agents provided it meets the quality control and performance requirements stated in the method.

SW-846 methods, with the exception of required method use for the analysis of method-defined parameters, are intended to be guidance methods which contain general information on how to perform an analytical procedure or technique which a laboratory can use as a basic starting point for generating its own detailed Standard Operating Procedure (SOP), either for its own general use or for a specific project application. The performance data included in this method are for guidance purposes only, and are not intended to be and must not be used as absolute QC acceptance criteria for purposes of laboratory accreditation.

Method EPA 200.7 and EPA 200.8 have been modified within the flexibility allowed in 40CFR136.6.

2.0 PRESERVATION AND BOTTLEWARE

All samples should be preserved with nitric acid to a pH of <2 at the time of collection. All sample pH are checked in sample receiving and within the metals department. Samples that are received with a pH >2 must be preserved to pH <2 and held for 24 hours prior to metals digestion to dissolve any metals that absorb to the container walls. Refer to SOP SAM101, current revision for further instruction. Final pH of TCLP extracts are checked and recorded in SGS Accutest - Orlando Extractions Department. Please refer to TCLP (1311) fluid determination logbook and SPLP (1312) fluid determination logbook for further information. TCLP extracts received from SGS Accutest - Orlando Extractions Department are prepared as soon as possible, no longer than 24 hours from time of receipt. If precipitation is observed during the sample preparation process the sample(s) are immediately re-prepped on dilution until no precipitation is observed. Samples received for dissolved metals analysis should be filtered and preserved to pH<2 as soon as possible and held for 24 hours prior to digestion. Refer to SGS Accutest - Orlando Sample Filtration Logbook for further information. All bottleware used by SGS Accutest - Orlando is tested

for cleanliness prior to shipping to clients. Analysis results must be < ½ RL to be acceptable. Refer to SOP SAM104, current revision for further instruction.

3.0 HOLDING TIME AND STORAGE

All samples should be digested and analyzed within 6 months of the time collection.

Aqueous samples do not require refrigeration.

4.0 REPORTING and METHOD DETECTION LIMITS

See analytical SOP MET100, current revision for further information.

5.0 INTERFERENCE

Organic substances in a matrix may cause interference if the sample is not digested rigorously enough. In addition, high levels of acids in the final digestate may cause interference in the analysis. This interference can be avoided by choosing the appropriate digestion method and by bringing the sample to an appropriate final volume. For a discussion of other interference, refer to specific analytical methods.

6.0 APPARATUS

The apparatus needed for this digestion procedure are listed below.

6.1 Automatic repipettor(s)

6.2 Fisher Brand 0.45 micron (um) filter or equivalent. Filter lots are checked for cleanliness through the Method Blank process. All Method Blank analytical results must be <1/2 RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated filters must be re-filtered through acceptable filters.

6.3 Environmental Express watch glasses or equivalent.

6.4 Thermometer(s)- capable of measuring a temperature of at least 125^0C, and checked against NIST traceable thermometers. Refer to SOP QA002, current revision for further information.

6.5 Environmental Express Hot Block or equivalent capable of maintaining a temperature of 90-95°C.

6.6 Environmental Express digestion vessels or equivalent, 65ml capacity. Each Lot of digestion tubes comes with a Certificate of Analysis which demonstrates cleanliness as well as volume accuracy at the 50ml mark. Please refer to Digestion Tube Certificate

Logbook for further information. Tube Lots are also checked through the Method Blank process. All Method Blank analytical results must be < ½ RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Re-digestion is required for all samples prepared with the contaminated tube lot.

6.7 Fisher Brand disposable 10 ml syringes or equivalent. Syringe lots are checked for cleanliness through the Method Blank process. All Method Blank results must be < ½ RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated syringes must be re-filtered through acceptable syringes.

6.8 Eppendorf Pipette (s) - Pipette (s) are checked daily for accuracy and to ensure they are in good working condition prior to use. Volumes are checked at 100% of maximum volume (nominal volume). Pipettes are checked within the metals department and results are stored electronically in the "Pipette Calibration Log". Refer to SOP QA006, current revision for further information regarding pipette calibration. BIAS: mean must be within 2% of nominal volume. Precision: RSD must be $\leq$ 1% of nominal volume based on three replicates.

6.9 Class A volumetric flask (s)

6.10 Class A volumetric pipette (s)

6.11 Class A graduated cylinder (s)

7.0 REAGENTS

All chemicals listed below are trace metal grade unless otherwise specified. Refer to Acid Certificate of Analysis logbook for Certificates of Analysis and compliance with the specifications of the grade listed. SGS Accutest - Orlando produces DI water to the specifications for the ASTM Type II standard designation based on system manufacturer's performance specifications. The DI water is used exclusively for laboratory purposes. Refer to SOP QA037, current revision for more information regarding testing and monitoring. De-ionized (DI) water should be used whenever water is required.

7.1 Hydrochloric acid. Fisher Trace metal grade or equivalent

7.2 Nitric acid. Fisher Trace metal grade or equivalent

7.3 Metals spiking solutions commercially purchased:

Environmental Express Multi-element spiking solution or equivalent made with 5% HNO3 and a trace of HF.

Inorganic Ventures 5000 mg/l Mineral solution or equivalent.

Prepared Metals standards:

100ppm Molybdenum, 100ppm Tin, 100ppm Strontium, and 100ppm Titanium spiking solution prepared as follows: Using a 10ml class A volumetric pipette, add 10mls of 1000ppm stock Molybdenum, 10mls of 1000ppm stock Tin, 10mls of 1000ppm stock Strontium, and 10mls of 1000ppm stock Titanium to a 100ml class A volumetric flask containing approximately 50mls of DI water and 3mls of concentrated Nitric acid and 5mls of concentrated HCL. Dilute to volume with DI water and mix well. This standard must be prepared every 6 months or before stock standard expiration date, whichever comes first. Refer to Metals Department Standard Prep Logbook for further information. Some of the information included in the logbook is as follows: standard name, elements in mix, manufacturer, lot number, parent expiration date, acid matrix, stock concentration, volume of standard added, total volume, final prepared concentration, prep date, initials, MET number, and prepared standard expiration date.

8.0 PROCEDURE

8.1 Shake sample vigorously to ensure thorough mixing. Measure out 50 ml of each sample into a labeled digestion vessel. The sample may be measured by using a Class A graduated cylinder or by using the calibrated digestion tube. Make sure that the sample identifications are accurately recorded on the digestion vessels and in the sample digestion log. In addition to the samples, a serial dilution (performed at the analytical bench), a post digestion spike (performed at the analytical bench), a matrix spike (MS), matrix spike duplicate (MSD), duplicate, blank spike and a method blank should be set up with each batch of 20 samples (10 samples for method 200.7). For the method blank and blank spike, 50 ml of DI water should be used. Refer to Table 1 for the spiking solution levels to use for each MS, MSD and blank spike.

8.1.1 When preparing TCLP samples use 5.0mls initial volume of extract and bring to a final volume of 50mls using DI water. Also prepare an additional leachate blank and leachate blank spike from the extraction fluid used for the samples. See section 8.8 and 8.9 of current METSOP 100 for acceptance criteria.

8.1.2 When preparing filtered samples for dissolved metals, an additional method blank must be prepared. This is performed to ensure there is no cross contamination from the filter media into the samples. The method blank must be filtered through the same filter media as the samples. See section 8.8 of current METSOP 100 for acceptance criteria.

8.2 Add 1.5 ml of concentrated nitric acid to all quality control and samples.

8.3 Pre heat the Hot Block to 90 to 95^{o}C. Place the labeled digestion vessels into the heating apparatus and cover with elevated or ribbed watch glasses. Heat the samples at a gentle reflux for 2 hours. Allow samples to cool.

8.4 Uncover all samples and matrix QC. Add an additional 1.5-ml of concentrated nitric acid to all quality control and samples. Continue heating, adding additional acid if necessary, until the digestion is complete, generally indicated when the digestate is light in color or does not change in appearance with continued refluxing. Allow samples to cool.

Note: If a client requires method 200.7 for the digestion, add 2.5ml of concentrated nitric acid instead of 1.5 ml in step 8.4. Continue heating the samples at a gentle reflux until the sample is completely digested. Signs of a complete digestion are if the digestate is light in color and/or if the appearance of the sample does not change with continued refluxing. More acid may be added as necessary to complete the digestion.

8.5 Add 2.5 ml of concentrated HCL to each sample and reflux for an additional 15 minutes. Allow samples to cool. Rinse digestion vessel walls with DI water.

Note: If a client requires method 200.7 for the digestion, add 7.5 ml of DI water along with the HCL.

8.6 Bring the samples to a final volume of 50.0 ml with DI water, cap and shake. The samples are now ready for analysis by ICP. If the sample contains particulate matter, it should be filtered along with the method blank and blank spike through a 0.45um syringe filter before analysis at the analytical bench.

8.7 Method EPA200.8 Digestion Procedure.

For the determination of total recoverable analytes in aqueous samples transfer a 50 mL aliquot from a well mixed, acid preserved sample to a labeled digestion vessel.

8.7.1 Pre heat hot block to a temperature of 90-95^0 C. Add 0.5 mL concentrated nitric acid and 0.25mL of concentrated hydrochloric acid to all samples and matrix QC digestion vessels containing the measured volume of sample. Place the samples along with all associated matrix QC on the hot block for solution evaporation and cover with elevated or ribbed watch glasses.

Note: For proper heating adjust the temperature control of the hot block such that an uncovered digestion vessel containing 50 mL of water placed in the center of the hot block can be maintained at a temperature approximately but no higher than 85°C. (Once the digestion vessel is covered with a watch glass the temperature of the water will rise to approximately 95°C.)

8.7.2 Gently heat the samples for 2 hours. DO NOT BOIL.

8.7.3 Allow the samples to cool. Bring to a final volume of 50mL using DI water, cap and shake. The samples are now ready for analysis.

8.7.4 Allow any undissolved material to settle overnight, or centrifuge a portion of the prepared sample until clear. (If after centrifuging or standing overnight the sample contains suspended solids that would clog the nebulizer, a portion of the sample may be filtered for their removal prior to analysis. However, care should be exercised to avoid potential contamination from filtration.) The MB and BSP must also be filtered in the same manner. All filtration is performed at the analytical bench.

9.0 QC REQUIREMENTS

For each digestion batch of 20 samples (10 samples per batch for method 200.7), a serial dilution (performed at analytical bench), a post digestion spike (performed at the analytical bench), a matrix spike, a matrix spike duplicate, a duplicate, a blank spike (LCS), and a method blank should be prepared. Re-digestion is suggested for QC that does not meet the SGS Accutest - Orlando QC limits. The appropriate lab supervisor or lab manager will notify the analyst of samples that need re-digestion. Please refer to TABLE 1 in this SOP for spiking volumes and concentrations. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements.

10.0 GLASSWARE CLEANING

All glassware should be washed with soap and tap water, rinsed with 5% nitric acid solution, and then rinsed at least 3 times with DI water. Refer to SOP GN196, current revision for further information regarding glassware cleaning.

11.0 DOCUMENTATION REQUIREMENTS

All digestion information should be documented in the Sample Digestion Logbook. The information required includes the sample identification (including the sample bottle number), the initial sample volume, and the final sample volume, the acids used (including lot number and manufacturer), the spiking solutions used, the digestion vessel lot number, the observed temperature, corrected temperature, the thermometer ID, analyst's signature, and the date of digestion. The analyst should write additional information such as unusual sample characteristics in the comment section.

12.0 SAFETY

The analyst should follow safety procedures as outlined in the SGS Accutest - Orlando Laboratory Safety Manual which includes the use of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acid contacts any part of the body, flush with water and contact the supervisor immediately.

13.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

13.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids must be followed. All method users must be familiar with the waste management practices described in Section 13.2.

13.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

14.0 GENERIC DEFINITIONS

14.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 24 hours whichever comes first.

14.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

14.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. A CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

14.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

14.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

14.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

14.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the performance of a method in a given sample matrix.

14.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and performance of a method in a given sample matrix.

14.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

14.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

14.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

15.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Statistical control limits are stored in the LIMS for QA purposes only. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

16.0 Hot Block Maintenance

Clean surface area of hot block periodically to prevent sample and reagent build up on the surface of the block. If the hot block cannot maintain a temperature between 90-95 degree C or the user experiences any other type of mechanical or electronic error a service representative will need to be contacted. Any hot block that is not functioning properly must be tagged as “Out of Service”.

Table 1: ICP Metals Spiking Levels
(Suggested levels, may vary depending on instrumentation used.)

ELEMENT	INITIAL CONC (ppm)	VOLUME USED (ml)	FINAL CONC (mg/l)	FINAL VOL. (ml)
Ba	200	0.50	2.0	50
Be	5	0.50	.05	50
Cd	5	0.50	.05	50
Cr	20	0.50	.20	50
Cu	25	0.50	.25	50
Co	50	0.50	0.50	50
Mn	50	0.50	0.50	50
V	50	0.50	0.50	50
Zn	50	0.50	0.50	50
As	200	0.50	2.0	50
Se	200	0.50	2.0	50
Pb	50	0.50	0.50	50
Tl	200	0.50	2.0	50
Sb	50	0.50	0.50	50
Mo	100	0.25	0.50	50
Sn	100	0.25	0.50	50
Al	200/5000	0.5/0.25	27	50
Fe	200/5000	0.5/0.25	26	50
Mg	5000	0.25	25	50
Ca	5000	0.25	25	50
K	5000	0.25	25	50
Na	5000	0.25	25	50
Ag	5	0.50	0.05	50
Ni	50	0.50	0.50	50
Sr	100	0.25	0.50	50
Ti	100	0.25	0.50	50

DIGESTION OF SOILS FOR ICP ANALYSIS

Prepared by:	David Metzgar III	Date:	06/21/2016
Approved by:	Svetlana Izosimova	Date:	06/21/2016

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TITLE: DIGESTION OF SOILS FOR ICP ANALYSIS

REFERENCES: 3050B

REVISED SECTIONS: SOP rebranded

1.0 SCOPE AND APPLICATION, SUMMARY

1.1 This method is applicable for the digestion of sediments, soils, sludges and solid wastes. After digestion, the samples can be analyzed by ICP. This digestion method is based upon SW846 method 3050B.

1.2 An aliquot of a homogenized soil is digested with repeated additions of nitric acid and hydrogen peroxide. Hydrochloric acid is added and the sample is refluxed for an additional 15 minutes. The sample is cooled to room temperature and diluted to 50 ml. If particulate matter is present, the sample is filtered.

2.0 PRESERVATION

All soils must be refrigerated at $\leq$ 6 ^{o}C. All bottleware used by SGS Accutest - Orlando is tested for cleanliness prior to shipping to clients. Analysis results must be < ½ RL to be acceptable. Please refer to SOP SAM104, current revision for further instruction.

3.0 HOLDING TIME

All samples should be digested and analyzed within 6 months of the time of collection.

4.0 INTEREFRENCES

Sludge and soil samples can contain diverse matrix types, which may contain a variety of interference. Spiked samples can be used to determine if this interference is adequately treated in the digestion process. For discussion of other interference, refer to specific analytical methods.

5.0 APPARATUS

The apparatus needed for this digestion procedure are listed below.

5.1 Automatic repipettor (s)

5.2 Fisher Brand 0.45 micron (um) filter or equivalent. Filter lots are checked for cleanliness through the Method Blank process. All Method Blank analytical results must be < ½ RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory

use. Samples filtered through the contaminated filters must be re-filtered through acceptable filters.

5.3 Top loader balance- capable of accurately weighing 0.01g. Refer to SOP QA005, current revision for balance calibration information.

5.4 Thermometer- capable of measuring to at least 125^0C and checked against NIST traceable thermometers. Refer to SOP QA002, current revision for further information.

5.5 Environmental Express Hot Block or equivalent capable of maintaining a temperature of 90-95°C.

5.6 Environmental Express digestion vessels or equivalent, 65ml capacity. Each Lot of digestion tubes comes with a Certificate of Analysis which demonstrates cleanliness as well as volume accuracy at the 50ml mark. Please refer to Digestion Tube Certificate Logbook for further information. Tube Lots are also checked through the Method Blank process. All Method Blank analytical results must be < ½ RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Re-digestion is required for all samples prepared with the contaminated tube lot.

5.7 Fisher Brand disposable 10 ml syringes or equivalent. Syringe lots are checked for cleanliness through the Method Blank process. All Method Blank results must be < ½ RL to be acceptable, if not, the contaminated lot must be identified and removed from laboratory use. Samples filtered through the contaminated syringes must be re-filtered through acceptable syringes.

5.8 Fisher Brand wooden spatulas or equivalent.

5.9 Eppendorf Pipette (s) - Pipette (s) are checked daily for accuracy and to ensure they are in good working condition prior to use. Volumes are checked at 100% of maximum volume (nominal volume). Pipettes are checked within the metals department and results are stored electronically in the “Pipette Calibration Log”. Refer to SOP QA006, current revision for further information regarding pipette calibration. BIAS: mean must be within 2% of nominal volume. Precision: RSD must be $\leq$ 1% of nominal volume based on three replicates.

5.10 Class A volumetric flask (s)

5.11 Class A volumetric pipette (s)

5.13 Teflon Chips

5.14 Solid Standard Reference Material (SRM) as required per project/client specific requirements.

5.15 Environmental Express ribbed watch glasses or equivalent.

6.0 REAGENTS

All chemicals listed below are trace metal grade unless otherwise specified. Refer to Acid Certificate of Analysis logbook for Certificate of Analysis and compliance with specifications of the grade listed. De-ionized (DI) water should be used whenever water is required. SGS Accutest Laboratories produces DI water to the specifications for the ASTM Type II standard designation based on the system manufacturer's performance specifications. The DI water is used exclusively for laboratory purposes. Refer to SOP QA037, current revision for more information regarding testing and monitoring.

6.1 Hydrochloric acid, Fisher Trace metal grade or equivalent

6.2 Nitric acid, Fisher Trace metal grade or equivalent

6.3 Hydrogen peroxide, reagent grade ,30%

6.4 Metals spiking solutions commercially purchased:

Environmental Express Multielement spiking solution or equivalent made with 5% HNO3 and a trace of HF.

Inorganic Ventures 5000 mg/l Mineral solution.

Prepared Metals Standards:

100ppm Molybdenum, 100ppm Tin, 100ppm Strontium, and 100ppm Titanium spiking solution prepared as follows: Using a 10ml class A volumetric pipette, add 10mls of 1000ppm stock Molybdenum, 10mls of 1000ppm stock Tin, 10mls of 1000ppm Strontium, and 10mls of 1000ppm Titanium to a 100ml class A volumetric flask containing approximately 50mls of DI water and 3mls of concentrated Nitric acid and 5mls of concentrated HCL. Dilute to volume with DI water and mix well. This standard must be prepared every 6 months or before stock standard expiration date, whichever comes first. Refer to Metals Standard Prep Logbook for further information. Some of the information included in the logbook is as follows: standard name, elements in mix, manufacturer, lot number, parent expiration date, acid matrix, stock concentration, volume of standard added, total volume, final prepared concentration, prep date, initials, MET number, and prepared standard expiration date.

7.0 PROCEDURE

7.1 Decant any free liquid from the solid sample. Remove any foreign objects such as twigs or rocks. The sample container must have enough room to move the matrix around with the wooden spatula. Mix the sample thoroughly using the wooden spatula. Make certain the entire sample is mixed well. The wooden spatula must reach the bottom of the original container and be able to be moved through the entire sample to ensure proper mixing. If the sample is packed tightly or matrix is dense and cannot be efficiently moved around in the original jar, a secondary container such as a porcelain dish must be used. Remove the sample from the original container and place in the clean secondary container. While in

the secondary container thoroughly mix sample around until appearing uniform in consistency. Upon completion the sample is re-packed into the original container. Refer to SOP QA034, current revision for more information on sample homogenization. Using a wooden spatula weigh out approximately 1.0 gram of a homogeneous sample on a top loading balance and place in the digestion vessel.

7.2 The sample identification must be accurately recorded on the digestion vessel and sample digestion log. In addition to the samples, a serial dilution (performed at the analytical bench), a post digestion spike (performed at the analytical bench), a matrix spike (MS), matrix spike duplicate (MSD), blank spike, duplicate (DUP) and a method blank should be set up with each batch of 20 samples. Refer to Table 1 for the spiking solution levels to use for each matrix spike, matrix spike duplicate, and blank spike. For the method blank and blank spike, 1.0 g of Teflon chips should be used. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements.

7.3 Add 2.5 ml of concentrated nitric acid to all quality control and samples. Cover all samples and matrix QC with ribbed watch glasses. Keep all samples covered throughout entire digestion procedure except during reagent additions.

7.4 Pre heat the Hot Block to 90 to 95^{o}C. Place the labeled digestion vessels into the heating apparatus. Heat the samples at a gentle reflux for 10-15 minutes at 90 to 95^{o}C. Allow the samples to cool.

7.5 Add an additional 2.5 ml of concentrated nitric acid to all quality control and samples. Heat the samples at a gentle reflux for an additional 30 minutes. Allow samples to cool.

> NOTE: If brown fumes are generated, which indicates oxidation of sample by HNO3, then repeat step 7.5 until no brown fumes are present.

7.6 Heat at 90 to 95°C without boiling until sample volume is reduced to approximately 2.5mls. Do not allow sample to go to dryness.

7.7 Allow samples to cool. Add 2 ml of DI water and 3 ml of 30% hydrogen peroxide to each sample and reflux until effervescence subsides.

7.6 Continue to add 30% hydrogen peroxide in 1ml aliquots with warming until the effervescence is minimal or until the general sample appearance is unchanged. Do not add more than a total of 10 mls of 30% hydrogen peroxide.

7.8 Heat at 90 to 95°C for 2 hours. Do not allow sample to go to dryness.

7.9 Allow samples to cool. Add 5 ml of concentrated HCl and reflux for an additional 15 minutes.

7.10 Allow the sample to cool. Dilute to final volume of 50 mls using DI water, cap and shake vessel. The sample is now ready for analysis by ICAP. If particulate matter is present, uncap the vessel and filter using Fisher Brand disposable syringe and 0.45 micron (um) filter or equivalent. The method blank and blank spike for the filtered sample's prep group must be filtered as well. All samples are filtered at the analytical bench.

8.0 QC REQUIREMENTS

For each digestion batch of 20 samples, a serial dilution (performed at the analytical bench), a post digestion spike (performed at the analytical bench), a matrix spike (MS), a matrix spike duplicate (MSD), a duplicate (DUP), a blank spike (LCS), and a method blank should be prepared. Re-digestion is suggested for QC that does not meet the SGS Accutest - Orlando QC limits. The appropriate lab supervisor or lab manager will notify the analyst of samples that need re-digestion. Please refer to TABLE 1 in this SOP for spiking volumes and concentrations. Refer to scheduling sheets and/or project specific QAPP for further information regarding client specific QC requirements.

9.0 GLASSWARE CLEANING

All glassware should be washed with soap and tap water and then soaked in a 5% nitric acid bath. It should then be rinsed at least 3 times with de-ionized water. Refer to SOP GN196, current revision for further information regarding glassware cleaning.

10.0 DOCUMENTATION REQUIREMENTS

All digestion information should be completed in the Metals Digestion Log. The information required includes: the sample identification (including bottle number), the initial sample weight, the final sample volume, the acids (including the lot number and manufacturer), the spiking solutions used, the observed temperature, the corrected temperature, the thermometer ID, the digestion vessel lot number, the filter lot number, the Teflon chips lot number, analysts signature, and the digestion date. The analyst should write additional information such as unusual sample characteristics in the comment section.

11.0 SAFETY

The analyst should follow normal safety procedures as outlined in the SGS Accutest - Orlando Laboratory Safety Manual which includes the uses of safety glasses and lab coats. In addition, all acids are corrosive and should be handled with care. Flush spills with plenty of water. If acids contact any part of the body, flush with water and contact supervisor.

12.0 POLLUTION PREVENTION AND WASTE MANAGEMENT

12.1 Pollution Prevention

Users of this method must perform all procedural steps in a manner that controls the creation and/or escape of wastes or hazardous materials to the environment. The amounts of standards, reagents and solvents must be limited to the amounts specified in this SOP. All safety practices designed to limit the escape of vapors, liquids or solids must be followed. All method users must be familiar with the waste management practices described in Section 12.2.

12.2 Waste Management

Individuals performing this method must follow established waste management procedures as described in the Sample and Laboratory Waste Disposal SOP SAM108, current revision. This document describes the proper disposal of all waste materials generated during the testing of samples.

13.0 GENERIC DEFINITIONS

13.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 24 hours whichever comes first.

13.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

13.3 Continuing Calibration Verification (CCV): A check standard used to verify instrument calibration throughout an analytical run. A CCV must be analyzed at the beginning of the analytical run, after every 10 samples, and at the end of the run.

13.4 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still be considered valid.

13.5 Initial Calibration (ICAL): A series of standards used to establish the working range of a particular instrument and detector. The low point should be at a level equal to or below the reporting level.

13.6 Initial Calibration Verification (ICV): A standard from a source different than that used for the initial calibration. A different vendor should be used whenever possible. The ICV is used to verify the validity of an Initial Calibration. This may also be called a QC check standard.

13.7 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

13.8 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the precision and bias of a method in a given sample matrix.

13.9 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

13.10 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

13.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

14.0 METHOD PERFORMANCE

Method performance is monitored through the routine analysis of negative and positive control samples. These control samples include method blanks (MB), blank spikes (BS), matrix spikes (MS), and matrix spike duplicates (MSD). The MB and BS are used to monitor overall method performance, while the MS and MSD are used to evaluate the method performance in a specific sample matrix.

Blank spike, matrix spike, and matrix spike duplicate samples are compared to method defined control limits. Statistical control limits are stored in the LIMS for QA purposes only. Additionally, blank spike accuracy is regularly evaluated for statistical trends that may be indicative of systematic analytical errors.

15.0 Hot block Maintenance

Clean surface area of hot block periodically to prevent sample and reagent build up on the surface of the block. If the hot block cannot maintain a temperature between 90-95 degree C or the user experiences any other type of mechanical or electronic error a service representative will need to be contacted. Any hot block that is not functioning properly must be tagged as “Out of Service”.

TABLE 1: ICP METALS SPIKING LEVELS
(Suggested levels, may vary depending on instrumentation used)

ELEMENT	INITIAL CONC (ppm)	VOLUME USED (ml)	FINAL CONC (mg/l)	FINAL VOL. (ml)
Ba	200	0.50	2.0	50
Be	5	0.50	.05	50
Cd	5	0.50	.05	50
Cr	20	0.50	.20	50
Cu	25	0.50	.25	50
Co	50	0.50	0.50	50
Mn	50	0.50	0.50	50
V	50	0.50	0.50	50
Zn	50	0.50	0.50	50
As	200	0.50	2.0	50
Se	200	0.50	2.0	50
Pb	50	0.50	0.50	50
Tl	200	0.50	2.0	50
Sb	50	0.50	0.50	50
Mo	100	0.25	0.50	50
Sn	100	0.25	0.50	50
Al	200/5000	0.5/0.25	27	50
Fe	200/5000	0.5/0.25	26	50
Mg	5000	0.25	25	50
Ca	5000	0.25	25	50
K	5000	0.25	25	50
Na	5000	0.25	25	50
Ag	5	0.50	0.05	50
Ni	50	0.50	0.50	50
Sr	100	0.25	0.50	50
Ti	100	0.25	0.50	50

APPENDIX A

1.0 Application

Appendix A designed to supplement SOPs MET104.xx and MET105.xx for the preparation of soil samples for compliance with DoD and certain state-specific projects

2.0 Background

A theory of particulate sampling was developed by geologist Pierre Gy to improve the quality of data gathered in support of mineral exploration and mining. The MIS approach described herein is based upon Gy's theories and is applicable to environmental sampling at contaminated sites.

A large portion of sampling error is a result of compositional and distributional heterogeneity.

Compositional heterogeneity describes the variability of contaminant concentrations between the particles that make up the population in the sample. This type of heterogeneity results in fundamental error (FE).

Distributional heterogeneity occurs when particles are not randomly distributed across the population due to slight spatial variations. Spatial variability will be missed if all samples are collected from one place. This type of heterogeneity results in grouping and segregation error (GSE).

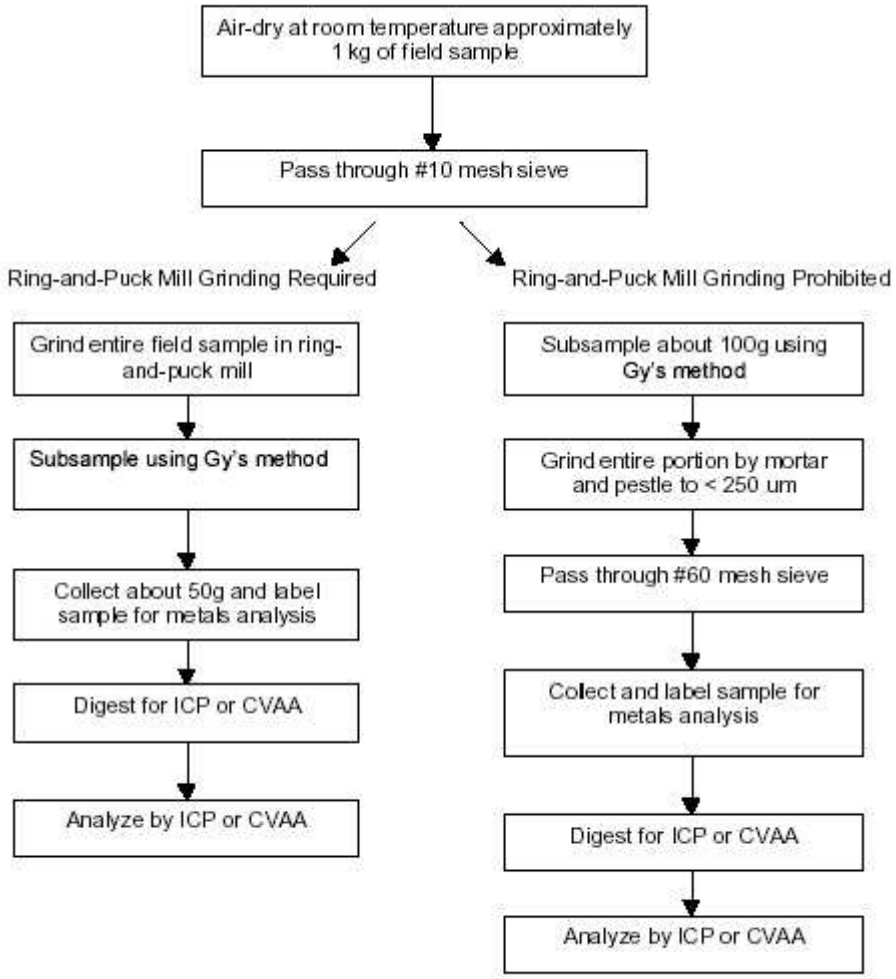
Gy found that fundamental error is directly proportionate to maximum particle size and inversely proportionate to sample size, therefore it is beneficial to collect and analyze a sample of sufficient size that consists of particulate matter where majority of contamination is present. In order to manage FE under 15%, particulate matter size must be under 2 mm and minimum sample mass above 30g.

To minimize GSE, it is imperative to collect sample increments randomly and in enough locations to capture the spatial variability, even within sample that already has been collected from the field.

3.0 Subsampling for Metals

Some projects require that metals analysis be performed on the multi-incremental sample that was collected for 8330B. The technique used should be listed in the project QAPP or SOW. Consult the client if this information is not available.

See flow chart below for various subsampling techniques:



If Ring and Puck Mill grinding is required, then proceed with the grinding procedure listed in SOP OP046 for explosives. The metallic components from the Ring and Puck Mill may introduce chromium and iron into the sample.

After grinding, place a baking tray on the downdraft table. Transfer the entire sample to the tray. Shape the sample into an elongated pile with flattened top surface that it is approximately 1 cm thick. Using a rectangular scoop, collect multiple top-to-bottom cuts across the sample (see figure below). A minimum of 4 cuts should be made through each sample. Combine the cuts in an appropriately labeled container. Minimum sample size should be 50 grams. Close the jar and repeat this procedure for each sample including the MB.

Transfer the samples to the metals department for analysis.

If Ring and Puck Mill grinding is not required then follow the procedure listed below.

Transfer the sample to a large ziplock bag after it has been air dried and sieved. Sample should be transferred over the downdraft tables to minimize dust contamination. Seal the bag and thoroughly mix the sample.

Place a baking tray on the downdraft table. Transfer the entire sample to the tray. Shape the sample into an elongated pile with flattened top surface that it is approximately 1 cm thick. Using a rectangular scoop, collect multiple top-to-bottom cuts across the sample (see figure below). A minimum of 4 cuts should be made through each sample. Combine the cuts in an appropriately labeled container. Minimum sample size should be 50 grams. Close the jar and repeat this procedure for each sample including the MB.

Return the remaining sample to the ziplock bag or mixing bowl.

Grind each sample and MB to a particle size less than 250 um with a non-metallic mortar and pestle.

Place a baking tray on the downdraft table. Sieve each sample through a #60 sieve onto a tray.

Collect and label the samples. Transfer the samples to the metals department for analysis.

For digestion withdraw approximately 5 g of sieved material. If mortar-and-pestle grinding was specified per QAPjP, 1 g is sufficient. Follow digestion procedure outlined in the body of this SOP.

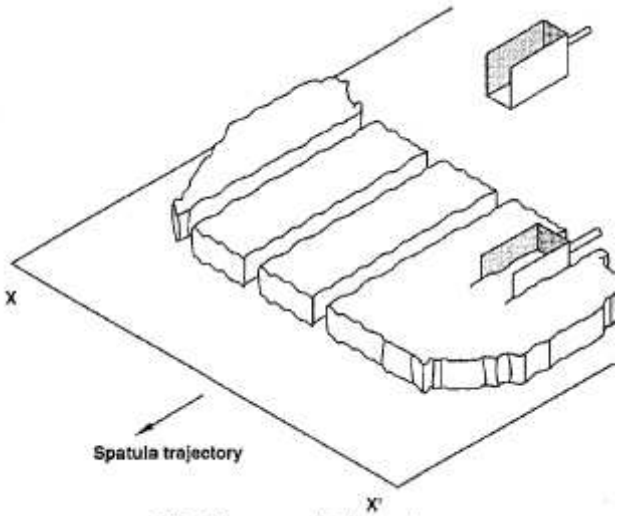


FIG. 1 Transversal Subsampling

STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF EXPLOSIVES (NITROAROMATICS, NITRAMINES AND NITRATE ESTERS) FROM WATER SAMPLES FOR HPLC ANALYSIS

Prepared by: Norm Farmer Date: 08/27/15

Approved by: Mike Eger Date: 09/04/15

Annual Review

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

Reviewed by: _____ Date: _____

Document Control

Issued to: QA Department Date: 09/09/15

Issued to: Organics Prep Department Date: *

Issued to: _____ Date: _____

Issued to: _____ Date: _____

Issued to: _____ Date: _____

Issued to: _____ Date: _____

Effective 7 days after "*" date

TEST NAME: STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF EXPLOSIVES (NITROAROMATICS, NITRAMINES AND NITRATE ESTERS) FROM WATER SAMPLES FOR HPLC ANALYSIS

Method: SW846 3535A/8330A, 3535A/8330B, and 3535A/8332

Dept: OP

Revised Sections: 10.0

1.0 Summary, Scope and Application

1.1 Summary

Aqueous samples are extracted using solid-phase extraction cartridges, eluted with acetonitrile, and stored in amber glass vials with Teflon lined screw caps.

1.2 Scope and Application

This procedure is applicable to aqueous samples submitted for explosives analysis by HPLC method SW-846 8330A, SW-846 8330B, and SW-846 8332. Samples for 8330A, 8332, and 8330B must be batched separately.

2.0 Discussion and Comments

This procedure is adapted from SW-846 methods 8330A, 8330B, 8332, 3500C and 3535A. The method utilizes “solid-phase extraction”; it is not applicable to samples requiring the “salting out” procedure. Samples expected to contain high levels of explosives should be screened using the direct injection technique.

Sample particulates may clog the solid-phase media and result in extremely slow sample extractions. Use of the appropriate filter aid may shorten the extractions without loss of analytes. Even when a filter aid is employed, this method may not be appropriate for samples high levels of suspended solids. It may be necessary to use smaller sample volumes with these types of samples. This should be noted on the sample prep sheet.

The HPLC detector is extremely sensitive and will respond to many organic compounds. It is important to minimize extraneous contaminants and carryover by scrupulously cleaning all glassware and by using only high purity reagents. Additionally, all extraction items that come in contact with the sample must be made from glass, stainless steel, or Teflon.

One of the current treatment processes for explosive site is the use of alkaline hydrolysis by treating the site with sodium hydroxide or calcium hydroxide. This may result in the samples having a high basic pH. If the samples are not neutralized prior to shipping to the lab, they should be neutralized prior to extraction. If the samples are not neutralized, the pH or the extract may also be high. This can damage the HPLC column.

3.0 Preservation and Holding Times

3.1 Preservation

3.1.1 Samples shall be collected in amber glass bottles with Teflon lined caps. One-liter bottles are recommended for aqueous samples.

3.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until extraction. The extracts must be refrigerated at ≤ 6°C until analysis.

3.2 Holding Time

3.2.1 Aqueous samples must be extracted within 7 days of collection. The Date/Time that the extraction is started and completed must be recorded on the prep sheet.

3.2.2 Extracts must be analyzed within 40 days of extraction.

4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples or 12 hours which ever comes first.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. Blank Spike Recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and are still considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the

analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Sample Duplicate (DUP): A replicate sample which is used to document the precision of a method in a given sample matrix.

4.8 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.9 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 Reagents

5.1 Acetonitrile – HPLC grade or equivalent

5.2 Methanol – HPLC grade or equivalent

5.3 RDX SPE Cartridges – Strata SDB or equivalent

5.4 Reagent water – HPLC grade or equivalent - free of interferences

5.5 Citric Acid, Crystalline – ACS grade or equivalent

5.6 Explosives **8330** Surrogate Solution – prepared in acetonitrile at a concentration specified by the HPLC analyst. All surrogate solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.7 Explosives **8330** Spike Solution – prepared in acetonitrile at a concentration specified by the HPLC analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.8 **8330 Plus** Spike Solution – (contains DNX, MNX, and TNX) - prepared in acetonitrile at a concentration specified by the HPLC analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.9 **PETN and NG (8332)** Spike Solutions – prepared in acetonitrile at a concentration specified by the HPLC analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

6.0 Glassware and Apparatus

6.1 Solid-phase cartridge extraction system – suitable for use with extraction cartridges

6.2 Vacuum pump

6.3 Connector with Teflon intake lines

6.4 10.0ml Receiver tubes

6.5 1000ml graduated cylinder

6.6 1.0 um glass fiber filters

6.7 Buchner Funnel

6.8 2000ml Vacuum Flasks

6.9 25ul, 250ul or 500ul syringes

6.10 pH paper

6.11 Disposable transfer pipette

6.12 Disposable 3.0ml syringes

6.13 0.45um Teflon syringe filters

6.14 2.0ml amber glass screw cap vials – caps must have Teflon lined septa

7.0 Procedure

7.1 The extraction of all samples must be documented on a "prep sheet". The prep sheet will include such items as: batch number, sample ID, bottle number, initial amount, final volume, solvent lot numbers, spike and surrogate lot numbers, batch numbers, extraction dates and times, and extraction technician.

The extraction technician is responsible for filling out all the required information on the prep sheet. A copy of the prep sheet will be submitted to the HPLC analyst with the extracts. The Batch number, extraction technician, and extraction start Date and Time are entered into LIMS.

7.2 Assemble the solid-phase extraction system. Label the side of each cartridge with the sample ID.

7.3 Wash each cartridge with 10 to 15 ml of acetonitrile. Use gravity flow if possible or a 1 to 2 ml/min flow rate. This will have to be done in 5ml aliquots. Do not allow the cartridge to go dry.

7.4 When only a small layer of acetonitrile remains above the sorbent bed, add 20 to 30 ml of reagent water. This will have to be done in 5ml aliquots. Do not allow the cartridge to go dry. Stop the flow just before the cartridge goes dry.

7.5 Fill each cartridge with about 5ml of reagent water.

7.6 Attach a connector and Teflon intake line to each of the cartridges.

7.7 Mark the level of the sample (upper edge) on the bottle with a marker.

7.8 Use 1 liter amber bottles for the method blank (MB) and blank spike (BS). Fill each of these bottles with 1000ml of reagent water.

7.9 Use a separate bottle for the matrix spike (MS) and the matrix spike duplicate (MSD). If there is only one bottle for the MS/MSD, it can be split into two aliquots. This should be noted on the prep sheet. Record the sample ID, bottle number, and volume on the prep sheet.

7.10 Samples that are extremely high in particulate may be pre-filtered through a 1.0 um glass fiber filter using a Buchner funnel and vacuum flask. If any samples are pre-filtered, then the MB must also be filtered.

7.11 Check the pH of each sample by dipping a disposable transfer pipette into the sample and touching it to the pH paper. Record the pH on the prep sheet. Samples for Explosives analysis generally should be extracted at the pH at which they were received; however, if the pH > 10 then the sample will need to be neutralized to prevent damage to the HPLC column. Adjust the pH to between 4 and 9 by adding small amounts of citric acid. Swirl the sample and recheck the pH after each aliquot is added.

7.12 Using the dedicated surrogate syringe add **250ul** of **8330 surrogate** solution to each of the samples including the QC samples. Record the surrogate lot number on the prep sheet.

7.13 Using the dedicated spike syringes add the appropriate spike solution(s) to the BS, MS, and MSD. Record the spike lot number on the prep sheet.

7.13.1 For 8330A only, add **250ul of 8330** spike solution to the BS, MS, and MSD.

7.13.2 For 8332 only, add **25ul of PETN and NG** spike solutions to the BS, MS, and MSD.

7.13.3 For 8330A and 8332, **250ul of 8330** and **25ul of PETN and NG** spike solutions to the BS, MS, and MSD.

7.13.4 For 8330A and LC+NG or LC+PETN, add **250ul of 8330** and **25ul of PETN and NG** spike solutions to the BS, MS, and MSD.

7.13.5 For 8330B, add **250ul of 8330** and **25ul of PETN and NG** spike solutions to the BS, MS, and MSD.

7.13.6 For LC+DNX, LC+MNX, and LC+TNX, add an additional **200ul of the 8330 Plus** spike solution to the BS, MS, and MSD.

7.14 Place the intake lines in the appropriate sample bottles.

7.15 Turn on the vacuum and draw the sample through the cartridge at a rate of about 10 ml/min, until the entire sample has passed through the cartridge. As particulate clogs the cartridge, increase the vacuum to maintain a reasonable flow rate.

7.16 Once the entire sample has been pulled through the cartridge, shut off the vacuum and remove the connector. Add 5ml of reagent water to the cartridge and turn on the vacuum. Draw the reagent water through the cartridge. Shut off the vacuum once the water has passed through the cartridge.

7.17 Fill each sample bottle to the sample mark with tap water. Transfer the water to a 1000ml graduated cylinder and record the sample volume. Discard the tap water.

7.18 Place an appropriately labeled 10.0ml receiver tube under each cartridge.

7.19 Add 2.5 to 3.0 ml of acetonitrile to each cartridge and allow it to pass through the cartridge under gravity flow. A slight vacuum may need to be applied if the cartridge media is clogged.

7.20 Adjust the volume to 10.0ml with reagent water and mix thoroughly.

7.21 (Optional) Transfer ~3ml of extract to disposable syringe. Attach a Teflon syringe filter to the disposable syringe. Filter the extract into an appropriately labeled amber 2.0ml screw cap vial.

CAUTION: WEAR SAFETY GLASSES, THE EXTRACT MAY SPRAY IF THE FILTER CLOGS.

7.22 Transfer the extracts to appropriately labeled amber 2ml screw cap vial. Store the extracts in the “extract refrigerator” until they are needed for analysis.

8.0 Quality Assurance and Quality Control

8.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. A batch may be held open for up to 12 hours; however, samples should not be added after the QC set has been completed. **NOTE:** Some project plans may require different batch definitions.

8.2 A method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD) must be extracted with each new batch of samples.

9.0 Safety and Waste Disposal

9.1 Safety

9.1.1 Safety glasses, gloves and lab coats should be worn when handling samples, standards or solvents.

9.1.2 Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS or SDS prior to using any new reagents or solvents.

9.1.3 Acetonitrile is an inhalation hazard and suspected carcinogen. Use in well ventilated area.

9.2 Waste Disposal

9.2.1 Waste acetonitrile is placed in the “non-chlorinated waste” container.

9.2.2 Spent solid-phase extraction cartridges may be disposed of in the trash.

9.2.3 Extracted water samples are rinsed down the drain with large amounts of water.

9.2.4 Samples are archived and stored for 30 days after analysis. After the storage time has elapsed, the remaining aqueous samples are transferred to the appropriate drums for disposal.

10.0 References

SW-846 Method 3500C, Rev. 3, 02/07

SW-846 Method 3535A, Rev. 1, 11/00

SW-846 Method 8330A, Rev. 1, 02/07

SW-846 Method 8332, Rev. 0, 12/96

SW-846 Method 8330B, Rev. 2, 10/06

STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF NITROAROMATICS AND NITRAMINES (EXPLOSIVES) FROM SOLID SAMPLES FOR HPLC ANALYSIS BY SW-846 8330B

Prepared by: (b) (6) Date: 09/21/15

Approved by: Date: 09/23/15

Annual Review

Reviewed by: Date:

Reviewed by: Date:

Reviewed by: Date:

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Issued to: Organics Prep Department Date: *

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Issued to: Date:

Effective 7 days after "*" date

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TEST NAME: **STANDARD OPERATING PROCEDURE FOR THE EXTRACTION OF NITROAROMATICS AND NITRAMINES (EXPLOSIVES) FROM SOLID SAMPLES FOR HPLC ANALYSIS BY SW-846 8330B**

Method: SW846 8330B

Dept: OP

Revised Sections: 3.2, 6.4, 6.7, 7.6, 7.13, 7.16, 7.17, 7.22, 7.23, 7.26, 8.2, 10.0, A2.0, A3.0, A4.0, A5.0, and A7.0

1.0 Summary, Scope and Application

1.1 Summary

Solid samples are air dried, sieved, and ground to a fine powder. Samples are then extracted with acetonitrile for 18 hours using a platform shaker table. The extracts are filtered and stored in amber glass vials with Teflon lined screw caps.

1.2 Scope and Application

This procedure is applicable to solid samples, including soils and sediments, submitted for Explosives analysis by HPLC method SW-846 8330B. For additional Department of Defense requirements see Appendix A. It is not applicable to samples submitted for analysis by SW-846 8330A.

2.0 Discussion and Comments

This procedure is adapted from SW-846 method 8330B. The method outlined in this SOP is designed for low and high concentration samples. Samples expected to contain high levels of explosives should be screened using method 8510, 8515 or other applicable methods. If the samples contain more than 2% explosives, they should not be ground.

Department of Defense contractors should screen all samples in the field for large pieces of explosive materials. Any large pieces of explosive material should be removed prior to sending the samples to the lab. However, the prep analysis should use caution when handling any samples for 8330B. If there are chunks of colored clay like material, large metal fragments, or any other suspicious material, contact the department supervisor immediately. Never grind any of this material without a thorough review.

The HPLC detector is extremely sensitive and will respond to many organic compounds. It is important to minimize extraneous contaminants and carryover by scrupulously cleaning all glassware, trays, and grinding equipment and by using only high purity reagents. Additionally, all extraction items that contact the sample should be made from glass, steel, wood, or Teflon.

3.0 Preservation and Holding Times

3.1 Preservation

3.1.1 This method utilizes multi-incremental sampling or the collection of large volume discreet samples. This can result in sample sizes of one to two kilograms. Samples shall be collected in heavy duty 1 or 2 gallon zip-lock bags. It is recommended that the samples be double bagged to prevent punctures. Smaller volume discreet samples may be collected in 250ml or 500ml amber glass jars with Teflon lined caps.

3.1.2 The samples must be protected from light and refrigerated at ≤ 6°C from the time of collection until drying. Once the samples have been air dried, they can be stored at room temperature. Samples should still be protected from light. The extracts must be refrigerated at ≤ 6°C until analysis.

3.2 Holding Time

3.2.1 Solid samples must be extracted within 14 days of collection. The Date/Time that the extraction is started and completed must be recorded on the prep sheet. **NOTE:** Extremely wet samples may take longer than the 14 day hold time to air dry. This must be communicated to the client.

3.2.2 Extracts should be analyzed as soon as possible but must be analyzed within 40 days of extraction.

4.0 Definitions

4.1 Batch: A group of samples which are similar with respect to matrix and the testing procedures being employed and which are processed as a unit. A sample batch is limited to a maximum of 20 samples that are extracted at the same time.

4.2 Blank Spike (BS): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. For this method, the spiked analytes are added after grinding. Blank Spike recoveries are used to document laboratory performance for a given method. This may also be called a Laboratory Control Sample (LCS).

4.3 Holding Time: The maximum times that samples may be held prior to preparation and/or analysis and still are considered valid.

4.4 Matrix Spike (MS): A sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. For this method, the spiked analytes are added after grinding. The matrix spike recoveries are used to document the bias of a method in a given sample matrix.

4.5 Matrix Spike Duplicate (MSD): A replicate sample aliquot spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. For this method, the spiked analytes are added after grinding. The matrix spike duplicate recoveries are used to document the precision and bias of a method in a given sample matrix.

4.6 Method Blank (MB): An analyte-free matrix to which all reagents are added in the same volumes or proportions as used in sample processing. The method blank is processed simultaneously with the samples through all the steps of the analytical procedure. The method blank is used to document contamination resulting from the analytical process.

4.7 Proficiency Test Sample (PT): An analyte-free matrix spiked with a known amount of analyte(s), processed simultaneously with the samples through all the steps of the analytical procedure. The PT sample is generally prepared by an outside vendor. This method requires that the PT sample go through the entire preparatory procedure including sieving and grinding. PT sample recoveries are used to document laboratory and method performance.

4.8 Sample Duplicate (DUP): A replicate sample taken after grinding which is used to document the precision of a method in a given sample matrix.

4.9 Sample Triplicate (TRP): A replicate sample taken after grinding which is used to document the precision of a method in a given sample matrix. DoD projects require the analysis of the sample triplicate.

4.10 Grinding Blank (GB): An aliquot of blank sand that is processed through the ring and puck mill between different samples. It is used to monitor for carry over between samples ground with the same bowl set.

4.11 Preservation: Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical integrity of the sample.

4.12 Surrogate: An organic compound which is similar to the target analyte(s) in chemical composition and behavior, but which is not normally found in environmental samples. Surrogates are used to measure the extraction efficiency.

5.0 Reagents

5.1 Acetonitrile – HPLC grade or equivalent

5.2 Methanol – HPLC grade or equivalent

5.3 Water – HPLC grade or equivalent

5.4 Blank Sand – precleaned to remove contaminants

5.5 Soil PT Sample – Environmental Resource Associates or Equivalent to meet DoD QSM requirements.

5.6 Explosives **8330** Surrogate Solution – prepared in acetonitrile at a concentration specified by the HPLC analyst. All surrogate solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.7 Explosives **8330** Spike Solution – prepared in acetonitrile at a concentration specified by the HPLC analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

5.8 Explosives **8332** Spike Solution – Contains PETN and NG and is prepared in acetonitrile at a concentration specified by the HPLC analyst. All spike solutions must be logged in the Spike and Surrogate Logbook and each solution must be verified prior to use.

6.0 Glassware and Apparatus

6.1 Aluminum baking trays – Half pan 18” x 13” or Full pan 18” x 26”

6.2 Aluminum foil and weigh dishes

6.3 Drying Cabinet

6.4 #10, #60, and #200 Brass or Stainless Steel Sieves

6.5 Spatula – stainless steel, wooden, or Teflon

6.6 Ring and Puck Mill

6.7 800cc bowls and pucks for Mill

6.8 0.5ml, 1.0ml, and 2.5ml syringes

6.9 4 ounce glass jars with Teflon lined caps

6.10 10.0-50.0ml repeat pipettor

6.11 Platform Shaker Table

6.12 Disposable 3.0ml or 5.0ml syringes

6.13 0.20um or 0.45um Teflon syringe filters

6.14 2.0ml amber glass screw cap vials – caps must have Teflon lined septa

6.15 Heavy Duty 1 and 2 gallon Zip-lock bags

6.16 Nitrile Gloves – shown to be interference free

6.17 Hi-Lo Thermometer

6.18 Downdraft Tables with Vacuum Filtration System

6.19 Top loading balance – capable of weighing samples to +/- 0.01 grams

6.20 Top loading balance – capable of weighing 2 kilogram samples to +/- 0.1 grams

7.0 Procedure

7.1 The extraction of all samples must be documented on a "prep sheet". The prep sheet will include such items as: batch number, sample ID, bottle number, initial amount, final volume, solvent lot numbers, spike and surrogate lot numbers, batch numbers, extraction dates and times, and extraction technician.

The extraction technician is responsible for filling out all the required information on the prep sheet. A copy of the prep sheet will be submitted to the HPLC analyst with the extracts. The Batch number, extraction technician, and extraction start Date and Time are entered into LIMS.

7.2 This method requires that the entire soil sample be processed prior to the final extraction procedure. Sample volumes received should typically be around 1 kilogram. The minimum sample volume that may be ground is 200 grams. If small sample volumes are received, or if there are additional analysis requested, notify the department supervisor. Department supervisor will need to confirm with Technical Director and/or Project Managers.

7.3 Label the side of an aluminum baking tray with the sample ID. Transfer the entire sample to tray, taking care to remove as much sample residue from the container as possible. Use a clean spatula and new pair of gloves for each sample. Spread the sample out to facilitate drying. Breakup any large clumps as much as possible. If the samples appear to contain a lot of clay, score the sample to facilitate breaking it into smaller pieces later.

7.4 Repeat this procedure for all of the samples in the batch. Prepare one tray with approximately 500 grams of clean sand. The amount of sand used for the method blank and blank spike should be similar to the mass of a typical sample that is ground in one aliquot. This will be dried, sieved and ground for use as the method blank (MB) and blank spike (BSP).

7.5 Samples must not be heated and should not be exposed to direct sunlight. Place the samples in the drying cabinet. Turn on the fans in the cabinet. Flow should be set high enough to keep air circulating, but not so high that sample would be drawn across the trays. Allow the samples to dry at room temperature, generally 2 to 5 days.

7.6 Record the room temperature in the appropriate logbook during each day of drying. If room temperature exceeds 26°C, notify the department supervisor. Department supervisor will need to notify the Technical Director and/or Project Managers who will notify the client.

7.7 A sample is generally considered dry when it is free flowing and any large clumps crumble easily. To test for dryness, transfer approximately 10 grams of sample to a labeled aluminum weigh dish. Weigh the sample and dish. Record this in the 8330B Air Drying Log. Return the sample and weigh dish to the drying cabinet. After a minimum of two hours, reweigh the sample and dish. If the weight did not change by more than 0.1 grams, the sample is considered dry.

7.7.1 If the weight changed by more than 0.1 grams, return the sample and weigh dish to the drying cabinet and check again after a minimum of two hours. If the weight did not change by more than 0.1 grams, the sample is considered dry.

7.7.2 If the weight is not constant after the 3rd weighing, the sample needs to dry longer. Repeat the process after and an additional 24 to 48 hours.

7.8 **NOTE**: The sieving and grinding procedures can generate a lot of dust. Utilize the downdraft tables when working with the samples to minimize dust transfer. Samples should be covered with aluminum foil or stored in the drying cabinet when not being processed. This reduces chances of cross contamination from other samples and degradation from light.

7.9 Once the samples are dry, transfer each one to an appropriately labeled mixing bowl. Record the weight of the entire sample to the nearest gram in the 8330B Weight Log.

7.10 The samples are sieved through a #10 stainless steel sieve. Breakup any clumps of soil with gloved hands (use a new pair of gloves for each sample). Do not intentionally include vegetation unless it is part of the project requirement. Excess vegetation should be stored with the portion that is unable to pass through the sieve.

NOTE: Samples with high clay content tend to form “bricks” when dried. It may not be possible to sieve this type of sample. Use the scored lines to break the sample into small pieces. The smaller pieces will be processed as if they had passed through the sieve. Note this in the prep log.

7.11 Collect and weigh any portion unable to pass through the sieve. Record the weight to the nearest gram. Record a brief description of the material. Store this fraction in a labeled zip-lock bag.

7.12 If samples are to be analyzed for metals prior to grinding, please see **Appendix A2.0** for the sub-sampling procedure.

CAUTION: DO NOT OPERATE RING AND PUCK MILL WITHOUT PROPER TRAINING.

7.13 Once the samples have been sieved, they must be ground to a fine powder. The 800cc can accommodate up to 600 gram sample aliquots. Do not overload the bowls, as this will decrease the grinding efficiency. Large sample volumes may require that multiple aliquots be ground. Use one bowl, puck and lid set for each sample. Do not use multiple sets per sample. Record the grinding order in the Grinding Log.

Specific Project Plans may require that the grinding step be skipped. This would be appropriate for production facilities or other sites were the contaminants were dispersed in a liquid form instead of a particulate form. For these samples, proceed to section 7.19.

7.14 **NOTE:** Do not grind aliquots less than 100 gram because this will cause excessive wear on the bowl and puck.

CAUTION: SAMPLES EXPECTED TO CONTAIN HIGH LEVELS OF EXPLOSIVES SHOULD BE SCREENED USING METHOD 8510, 8515 OR OTHER APPLICABLE METHODS. IF THE SAMPLES CONTAIN MORE THAN 2% EXPLOSIVES, THEY SHOULD NOT BE GROUND.

7.15 Process one sample at a time. Place the appropriate puck in the appropriate bowl. Transfer 500 to 600 grams of a sample to the bowl. Place the appropriate lid on the bowl. Use the pneumatic lift to load the bowl into the mill. Record the grinding order in the Grinding Log.

7.16 Close the lid to the mill. The mill will not operate with the lid open. Press and hold the green start button. Once the mill starts, release the button. The mill is programmed to run for 1 minute. Samples suspected of containing crystalline energetic residues (TNT, RDX, HMX, and their breakdowns) can be adequately ground using 2 one minute cycles. Samples suspected of containing polymeric residues (propellants and nitrocellulose) can be adequately ground using 5 one minute cycles. Accutest grinds all samples for a minimum of 5 one minute cycles to ensure adequate grinding regardless of residue type. Allow the sample and bowl to cool between each grind. See **Appendix A3.0** for grinding temperature study.

7.17 Repeat the grinding procedure for each sample aliquot until the entire sample has been processed. The bowl, puck and lid must be thoroughly cleaned between different samples; however, it is not necessary to clean the bowl, puck and lid between multiple aliquots of the same sample.

The bowls, pucks, and lids are cleaned with water and detergent solution. Brushes are used to facilitate the process. Particular attention must be paid to the grinding edges and handle of the puck. The bowls, pucks, and lids are then rinsed with tap water and DI water. The bowls, pucks, and lids can not be allowed to air dry. The low chrome content of material will cause surface rusting.

The bowls, pucks, and lids should be rinsed with methanol and then dried with disposable towels.

See **Appendix A4.0** for additional DoD requirements for the preparation of grinding blanks.

7.18 Document the sample ID, bowl set, and grinding order in the appropriate logbook.

7.19 Transfer all of the aliquots of a sample to a large zip-lock bag. Sample should be transferred over the downdraft tables to minimize dust contamination. Seal the bag and thoroughly mix the sample.

7.20 Place a baking tray on the downdraft table. Spread out the sample on a baking tray so that it is approximately 1 cm thick.

7.21 Using a spatula, collect at least 30 different increments (~0.3 gram each) from randomly chosen locations in the sample. Combine the increments in an appropriately labeled 4 ounce jar. Nominal sample size should be 10 grams. Record the weight to the nearest 0.01 gram on the prep sheet.

7.22 Close the jar and repeat this procedure for each sample including the QC samples. This includes the method blank (MB), blank spike (BS), matrix spike (MS), and matrix spike duplicate (MSD) and duplicate (DUP). Use 10.0 gram aliquots of clean sand for the MB and BS. Use additional 10.0 gram aliquots of a sample for the MS, MSD, DUP, and TRP. Record the sample ID, bottle number, and weight on the prep sheet.

7.23 DoD projects require the analysis of a proficiency test sample (PT) per batch. **NOTE:** If the samples do not require grinding, then the PT sample does not need to be extracted or analyzed. See **Appendix A5.0**.

The PT sample differs from the blank spike in two ways. An outside vendor generally prepares the PT sample. The PT sample must be processed through all preparatory steps, including grinding. However, the PT sample should not be air dried; this would result in the loss of the more volatile components.

The PT sample should be ground in the same manner as the samples. The ground PT should be stored in a sealed glass jar. Generally the PT sample is ground when it is first opened and then sub-sampled for multiple batches. It is cost prohibitive to grind a fresh PT sample with each batch of samples; however, some project plans may require that a fresh PT sample be prepared for the project.

7.24 Using the dedicated spike syringe add 1.25ml of 8330 and 8332 spike solution to the BS, MS, and MSD. Record the spike lot number on the prep sheet.

7.25 Using the dedicated surrogate syringe add 1.25ml of 8330 surrogate solution to each of the samples including the QC samples. Record the surrogate lot number on the prep sheet.

7.26 Using a graduated pipettor or cylinder, add **18.75ml** of acetonitrile or each of the sample vials, the method blank (MB), sample duplicate (DUP), and sample triplicate (TRP). Add **16.25ml** of acetonitrile to the BS, MS, and MSD. This will result in **20.0ml** of acetonitrile in each of the jars.

7.27 Put the cap on each jar and shake briefly to mix.

7.28 Place the jars in the covered rack on the platform shaker.

7.29 Shake the samples at a rate of 100 rpm for 18 hours. After 18 hours, turn off the shaker and remove the jars.

7.30 Using a repeat pipettor add 30.0ml of water to each of the samples including the QC samples.

7.31 Put the cap on each jar and shake briefly to mix. This results in a final volume of 50.0 ml.

7.32 Transfer 3-5ml of extract to disposable syringe. Attach a Teflon syringe filter to the disposable syringe.

7.33 Filter the extract into appropriately labeled amber 2.0ml screw cap vial.

CAUTION: WEAR SAFETY GLASSES, THE EXTRACT MAY SPRAY IF THE FILTER CLOGS.

7.34 Store the extracts in the “extract refrigerator” until they are needed for analysis.

8.0 Quality Assurance and Quality Control

8.1 An extraction batch is defined as samples of a similar matrix that are prepared for a particular parameter. The batch size is limited to 20 samples. Samples can not be added to the batch after the grinding procedure has started.

8.2 A method blank (MB), blank spike (BS), matrix spike (MS), matrix spike duplicate (MSD), duplicate (DUP), triplicate (TRP), and ground PT sample must be extracted with each new batch of samples.

8.3 Additional QA/QC requirements are listed in Appendix A.

9.0 Safety and Waste Disposal

9.1 Safety

9.1.1 Safety glasses, gloves and lab coats should be worn when handling samples, standards or solvents.

9.1.2 Avoid grinding samples that may contain high levels of explosives. The grinding action may cause them to DETONATE.

9.1.3 **Hearing protection** should be worn while operating the ring and puck mill.

9.1.4 Material Safety Data Sheets (MSDS) or Safety Data Sheets (SDS) are available for all reagents and solvents used in the lab. Technicians should review the MSDS or SDS prior to using any new reagents or solvents.

9.1.5 Acetonitrile is an inhalation hazard and suspected carcinogen. Use in well ventilated area.

9.1.6 The fine dust created during the drying, sieving, and grinding procedures is an inhalation hazard. Fume hoods or downdraft tables should be used to minimize exposure to dust.

9.2 Waste Disposal

9.2.1 Waste acetonitrile is placed in the “non-chlorinated waste” container.

9.2.2 Extracted soil samples and residual acetonitrile may be poured into the “non-chlorinated waste” container or the entire jar may be lab packed with the “extract waste”.

9.2.3 The remaining processed soil samples and material that did not pass through the #10 sieve should be bagged, labeled, and stored until time of disposal. **NOTE:** Soils from foreign soils must follow additional “foreign soil” disposal requirements.

9.2.4 Samples are archived and stored for 30 days after analysis. After the storage time has elapsed, the remaining soil samples are transferred to the appropriate drums for disposal.

10.0 References

SW-846 Method 8330A, Rev. 1, 02/07

SW-846 Method 8330B, Rev. 2, 10/06

DoD Quality Systems Manual Version 4.2, October 2010

DoD Quality Systems Manual Version 5.0, July 2013

Extraction Kinetics of Energetic Compounds from Training Range and Army Ammunition Plant Soils: Platform Shaker versus Sonic Bath Methods, M.E. Walsh and D.J. Lambert, ERDC/CRREL TR06-6, 2006

DoD Environmental Data Quality Workgroup, Guide for Implementing EPA SW-846 Method 8330B, July 2008

DoD Environmental Data Quality Workgroup, DoD Training – ELAP Requirements for 8330B, December 2014

Standard Guide for Laboratory Subsampling of Media Related to Waste Management Activities, ASTM D 6323, 1998 (Re-approved 2003)

ESSA Mill Instruction Manual

APPENDIX A

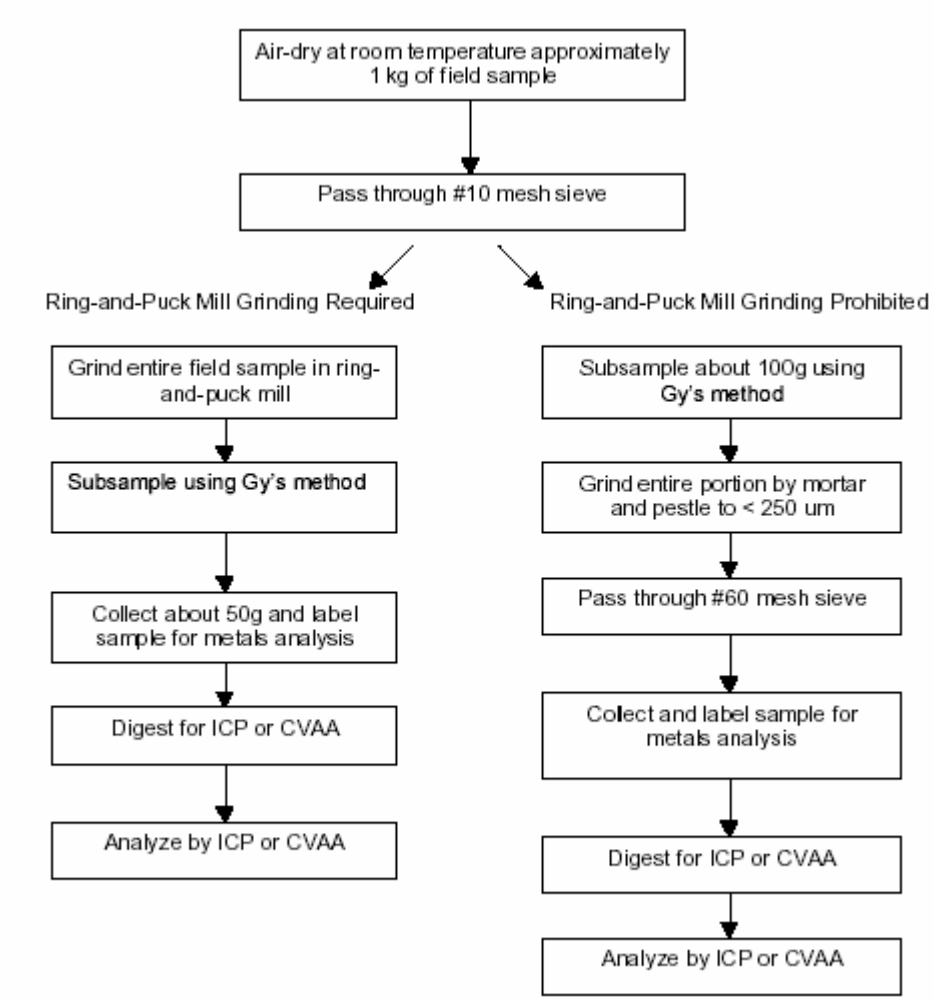
A1.0 Application

Appendix A is designed to supplement SOP OP046.x for the preparation of soil samples for SW-846 8330B. This appendix outlines additional requirements for compliance with Department of Defense QSM 4.2 and 5.0 projects.

A2.0 Sub-sampling for Metals

Some projects require that metals analysis be performed on the multi-incremental sample that was collected for 8330B. The technique used should be listed in the project QAPP or SOW. Consult the client if this information is not available.

See flow chart below for various subsampling techniques:



If Ring and Puck Mill grinding is required, then proceed with the grinding procedure listed in this SOP for explosives. The metallic components from the Ring and Puck Mill may introduce chromium and iron into the sample.

After grinding, place a baking tray on the downdraft table. Transfer the entire sample to the tray. Shape the sample into an elongated pile with flattened top surface that it is approximately 1 cm thick.

Use a spatula to collect 30-50 different increments (~1.0 gram each) from randomly chosen locations in the sample. Combine the increments in an appropriately labeled container. Minimum sample size should be 50 grams. Close the jar and repeat this procedure for each sample including the MB.

Alternatively

Use a rectangular scoop to collect multiple top-to-bottom cuts across the sample (see figure 1 below). A minimum of 4 cuts should be made through each sample. Combine the cuts in an appropriately labeled container. Minimum sample size should be 50 grams. Close the jar and repeat this procedure for each sample including the MB.

Transfer the samples to the metals department for analysis.

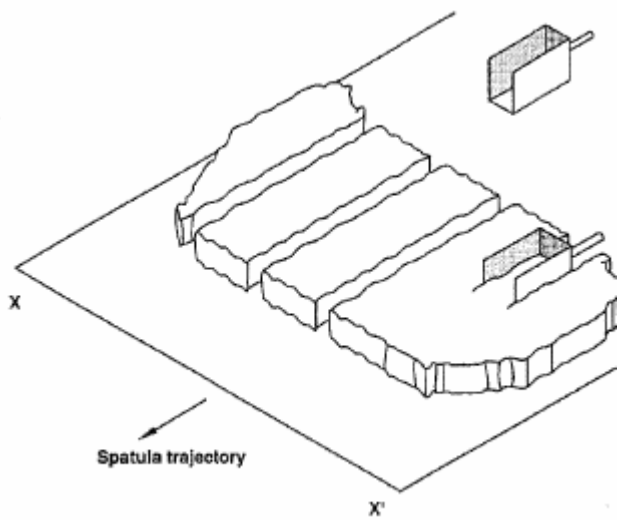


FIG. 1 Transversal Subsampling

If grinding is to be done by mortar and pestle, then follow the procedure listed below.

Transfer the air dried and sieved sample to a large zip-lock bag. Seal the bag and thoroughly mix the sample.

Place a baking tray on the downdraft table. Transfer the entire sample to the tray. Shape the sample into an elongated pile with flattened top surface that it is approximately 1 cm thick.

Use a spatula to collect 30-50 different increments (~1.0 gram each) from randomly chosen locations in the sample. Combine the increments in an appropriately labeled container. Minimum sample size should be 50 grams. Close the jar and repeat this procedure for each sample including the MB.

Alternatively

Use a rectangular scoop to collect multiple top-to-bottom cuts across the sample (see figure 1 below). A minimum of 4 cuts should be made through each sample. Combine the cuts in an appropriately labeled container. Minimum sample size

should be 50 grams. Close the jar and repeat this procedure for each sample including the MB.

Return the remaining sample to the zip-lock bag or mixing bowl.

Grind each sample and MB to a particle size less than 250 um with a non-metallic mortar and pestle. Collect and label the samples.

Transfer the samples to the metals department for analysis.

If additional sieving is required, then follow the procedure listed below.

In section 7.10, place a #60 sieve after the #10 sieve. Specific projects may require alternate sieve sizes. Sieve the sample through the #10 and #60 sieves.

Collect and weigh any portion unable to pass through each sieve. Record the weight to the nearest gram. Record a brief description of the material. Store this fraction in a labeled zip-lock bag. The aliquot that passed through the #60 is generally what will be analyzed.

Transfer the air dried and sieved sample to a large zip-lock bag. Seal the bag and thoroughly mix the sample.

Place a baking tray on the downdraft table. Transfer the entire sample to the tray. Shape the sample into an elongated pile with flattened top surface that it is approximately 1 cm thick.

Use a spatula to collect 30-50 different increments (~1.0 gram each) from randomly chosen locations in the sample. Combine the increments in an appropriately labeled container. Minimum sample size should be 50 grams. Close the jar and repeat this procedure for each sample including the MB.

Alternatively

Use a rectangular scoop to collect multiple top-to-bottom cuts across the sample (see figure 1 below). A minimum of 4 cuts should be made through each sample. Combine the cuts in an appropriately labeled container. Minimum sample size should be 50 grams. Close the jar and repeat this procedure for each sample including the MB.

Return the remaining sample to the zip-lock bag or mixing bowl.

Collect and label the samples. Transfer the samples to the metals department for analysis.

A3.0 Sample Grinding Cycles

Samples for DoD projects require that all samples be ground for **5** one minute cycles. Samples should be allowed to cool for at least two minutes between cycles. DoD guidelines originally require that the puck be removed from the sample between grinding cycles; however, this is not necessary since the heat generated is far less than expected. Results of an in-house temperature study are shown in Table 1:

TABLE 1

4/24/2008	Sample at Puck Grinding Area	Lid	Outer Bowl at Contact Area
Starting Temperature	22.4	22.8	22.8
After 1st minute	24.6	23.6	23.8
After 2nd minute	24.8	23.4	25.2
After 3rd minute	25.4	23.6	25.2
After 4th minute	25.8	24.2	25.2
After 5th minute	26.0	24.6	25.2
Temperatures in Degree C by IR thermometer.			
At the end of each minute, the grinding bowl was removed from the ring and puck mill to measure the temperature of the lid and outer bowl, and then opened briefly to measure the temperature of the sample at the puck. Total time between grinds was approximately 15 seconds.			

A4.0 Cleaning Process and Grinding Blanks

The aluminum baking trays, mixing bowls, stainless steel sieves and grinding equipment must be completely cleaned between samples. The trays, bowls, and sieves are cleaned with water and detergent solution. Brushes are used to facilitate the process. The trays, bowls, and sieves are then rinsed with tap water and DI water. These items are allowed to air dry or they can be rinsed with methanol to speed up the drying process.

The bowls, pucks, and lids are cleaned in a similar fashion. Particular attention must be paid to the grinding edges and handle of the puck. It is not necessary to clean the bowl, puck and lid between multiple aliquots of the same sample. The bowls, pucks, and lids

can not be allowed to air dry. The low chrome content of material will cause surface rusting. The bowls, pucks, and lids should be rinsed with methanol and then dried with disposable towels.

Bowls, pucks, and lids are numbered as sets. In order to properly track and assess potential cross-contamination, the bowl sets must not be mixed. The grinding order must be documented and a grinding blank must be prepared on each bowl set prior to use and between each sample. It is not necessary to prepare a grinding blank between multiple aliquots of the same sample. See Grinding Log for numbering instructions.

Grinding blanks for each Ring and Puck set should be prepared before the first sample and after the last sample (not to exceed 10 samples).

To prepare a grinding blank, place the appropriate puck in the appropriate bowl. Transfer a volume of blank sand similar to that used for the samples (200-500 grams) to the bowl. Place the appropriate lid on the bowl. Use the pneumatic lift to load the bowl into the mill. Record the grinding order in the Grinding Log.

Close the lid to the mill. The mill will not operate with the lid open. Press and hold the green start button. Once the mill starts, release the button. The mill is programmed to run for 1 minute. The grinding blank can be adequately ground using 2 one minute cycles.

Transfer the grinding blank to an appropriately labeled zip-lock bag. This should be transferred over the downdraft tables to minimize dust contamination. Seal the bag and thoroughly mix the sample.

The grinding blanks for each Ring and Puck Set can be sub-sampled and composited into a single sample to prior to extraction. This would result in a maximum of 3 grinding blanks if all 3 Ring a Puck sets were used.

A5.0 Proficiency Test Sample Requirements

Samples for DoD projects require an additional proficiency test (PT) sample. The PT sample differs from the blank spike in two ways. An outside vendor generally prepares the PT sample. The PT sample must be processed through all preparatory steps, including grinding. However, the PT sample should not be air dried; this will result in the loss of the more volatile components.

The PT sample is generally supplied as a 500gram sample. The PT may be split into 2 x 250gram aliquots. If the PT is split, store the second aliquot in a sealed glass jar at ≤ 6°C until needed.

The PT sample should be ground in the same manner as the samples. The ground PT should be stored in a sealed glass jar at ≤ 6°C. Generally the PT sample is ground when it is first opened and then sub-sampled for multiple batches. It is cost prohibitive to grind a fresh PT sample with each batch of samples; however, some project plans may require that a fresh PT sample be prepared for the project.

NOTE: The PT sample may not contain all analytes of interest for a given project. If additional analytes are required, they must be spiked into the PT sample prior to grinding.

A6.0 Grinding Demonstration

The adequacy of the grinding by the ring and puck mill can be checked by periodically placing a small aliquot of a grinding blank on a #200 sieve and checking to see if the material passes through the sieve. Since this is a 75um sieve, it would take a long time to process a large aliquot.

A7.0 Ring and Puck Mill Maintenance

Refer to equipment manual for details on scheduled maintenance items. **NOTE**: If the Ring and Puck mill is not being used weekly then the Daily, Weekly, and Monthly Maintenance can be extended.

Daily Maintenance

Press drain buttons to expel any water in pneumatic lines.

Check air pressure supply is a minimum of 80 psi.

Weekly Maintenance

Check floor mounting bolts for signs of corrosion or fatigue.

Inspect bowl lid o-rings for signs of wear. Replace o-rings as needed.

Monthly Maintenance

Inspect platform insert for signs of wear on load bearing surfaces. Replace insert when wear exceeds 2mm.

Empty dust tray.

Other Maintenance

Lubricate motor plate bearings and drive shaft every 150 hours of use.

Check belt tension every 150 hours of use.

SGS Savannah Limits

	Units	DL(MDL)	LOD	LOQ (RL)
Aluminum	mg/kg	1.75	2.5	10
Antimony	mg/kg	0.065	0.25	1
Arsenic	mg/kg	0.1	0.25	0.5
Barium	mg/kg	0.05	0.1	10
Beryllium	mg/kg	0.025	0.05	0.25
Cadmium	mg/kg	0.025	0.05	0.2
Calcium	mg/kg	2.5	5	250
Chromium	mg/kg	0.05	0.1	0.5
Cobalt	mg/kg	0.025	0.05	2.5
Copper	mg/kg	0.05	0.1	1.25
Iron	mg/kg	0.85	2.5	15
Lead	mg/kg	0.05	0.2	1
Magnesium	mg/kg	1.8	5	250
Manganese	mg/kg	0.025	0.05	0.75
Molybdenum	mg/kg	0.025	0.05	2.5
Nickel	mg/kg	0.025	0.05	2
Potassium	mg/kg	10	25	500
Selenium	mg/kg	0.12	0.25	1
Silver	mg/kg	0.041	0.1	0.5
Sodium	mg/kg	25	100	500
Strontium	mg/kg	0.025	0.05	0.5
Thallium	mg/kg	0.055	0.25	0.5
Tin	mg/kg	0.045	0.05	2.5
Titanium	mg/kg	0.025	0.1	0.5
Vanadium	mg/kg	0.025	0.05	2.5
Zinc	mg/kg	0.15	0.25	1
Mercury	mg/kg	0.0042	0.017	0.042

	Units	DL(MDL)	LOD	LOQ (RL)
Aluminum	ug/l	14.0	25.0	200
Antimony	ug/l	1.0	5.0	6.0
Arsenic	ug/l	1.3	5.0	10.0
Barium	ug/l	1.0	5.0	200
Beryllium	ug/l	0.2	1.0	4.0
Cadmium	ug/l	0.2	1.0	5.0
Calcium	ug/l	50	100	1000
Chromium	ug/l	1.0	5.0	10.0
Cobalt	ug/l	0.2	1.0	50.0
Copper	ug/l	1.0	2.0	25.0
Iron	ug/l	17.0	50.0	300
Lead	ug/l	1.1	2.0	5.0
Magnesium	ug/l	35	100	5000
Manganese	ug/l	1.0	2.0	15.0
Molybdenum	ug/l	0.3	2.0	50.0
Nickel	ug/l	0.4	1.0	40.0
Potassium	ug/l	200	500	10000
Selenium	ug/l	2.9	5.0	10.0
Silver	ug/l	0.7	2.0	10.0
Sodium	ug/l	500	2000	10000
Strontium	ug/l	0.5	1.0	10.0
Thallium	ug/l	1.4	2.0	10.0
Tin	ug/l	1.0	2.0	50.0
Titanium	ug/l	1.0	2.0	10.0
Vanadium	ug/l	0.6	2.0	50.0
Zinc	ug/l	4.4	5.0	20.0
Mercury	ug/l	0.03	0.1	0.5

TCLP	Units	DL(MDL)	LOD	LOQ (RL)
Aluminum	mg/l	0.14	0.25	2
Antimony	mg/l	0.01	0.05	0.06
Arsenic	mg/l	0.013	0.05	0.1
Barium	mg/l	0.05	0.05	2
Beryllium	mg/l	0.002	0.01	0.04
Cadmium	mg/l	0.002	0.01	0.05
Calcium	mg/l	0.5	1	10
Chromium	mg/l	0.01	0.05	0.1
Cobalt	mg/l	0.002	0.01	0.5
Copper	mg/l	0.01	0.02	0.25
Iron	mg/l	0.17	0.5	3
Lead	mg/l	0.011	0.02	0.05
Magnesium	mg/l	0.35	1	50
Manganese	mg/l	0.01	0.02	0.15
Molybdenu	mg/l	0.003	0.02	0.5
Nickel	mg/l	0.004	0.01	0.4
Potassium	mg/l	2	5	100
Selenium	mg/l	0.029	0.05	0.1
Silver	mg/l	0.007	0.02	0.1
Sodium	mg/l	5	20	100
Strontium	mg/l	0.005	0.01	0.1
Thallium	mg/l	0.014	0.02	0.1
Tin	mg/l	0.01	0.02	0.5
Titanium	mg/l	0.01	0.02	0.1
Vanadium	mg/l	0.006	0.02	0.5
Zinc	mg/l	0.10	0.10	0.20
Mercury	mg/l	0.0003	0.001	0.005

TA Denver Limits - Solids

Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
6010C_DOD5	3050B	Aluminum	7429-90-5	50.0	1.55	6.00	mg/Kg	74	119	20	74	119	20		
		Antimony	7440-36-0	2.00	0.733	1.50	mg/Kg	79	114	20	79	114	20		
		Arsenic	7440-38-2	2.50	0.665	2.50	mg/Kg	82	111	20	82	111	20		
		Barium	7440-39-3	2.00	0.104	0.300	mg/Kg	83	113	20	83	113	20		
		Beryllium	7440-41-7	0.500	0.0330	0.120	mg/Kg	83	113	20	83	113	20		
		Cadmium	7440-43-9	0.500	0.0410	0.150	mg/Kg	82	113	20	82	113	20		
		Calcium	7440-70-2	100	14.1	50.0	mg/Kg	81	116	20	81	116	20		
		Chromium	7440-47-3	3.50	0.0580	0.200	mg/Kg	85	113	20	85	113	20		
		Cobalt	7440-48-4	1.00	0.100	0.400	mg/Kg	85	112	20	85	112	20		
		Copper	7440-50-8	5.00	0.217	0.800	mg/Kg	81	117	20	81	117	20		
		Iron	7439-89-6	80.0	3.80	15.0	mg/Kg	81	118	20	81	118	20		
		Lead	7439-92-1	0.900	0.310	0.800	mg/Kg	81	112	20	81	112	20		
		Magnesium	7439-95-4	30.0	3.70	14.0	mg/Kg	78	115	20	78	115	20		
		Manganese	7439-96-5	4.50	0.100	0.400	mg/Kg	84	114	20	84	114	20		
		Nickel	7440-02-0	4.00	0.132	0.450	mg/Kg	83	113	20	83	113	20		
		Potassium	7440-09-7	300	41.0	160	mg/Kg	81	116	20	81	116	20		
		Selenium	7782-49-2	3.00	0.860	3.00	mg/Kg	78	111	20	78	111	20		
		Silver	7440-22-4	1.50	0.160	0.600	mg/Kg	82	112	20	82	112	20		
		Sodium	7440-23-5	500	59.0	200	mg/Kg	83	118	20	83	118	20		
		Strontium	7440-24-6	1.00	0.0360	0.100	mg/Kg	83	114	20	83	114	20		
		Thallium	7440-28-0	3.00	0.650	2.50	mg/Kg	83	111	20	83	111	20		
		Vanadium	7440-62-2	2.00	0.0940	0.350	mg/Kg	82	114	20	82	114	20		
		Zinc	7440-66-6	8.00	0.398	1.50	mg/Kg	82	113	20	82	113	20		

8330B_DOD5	8330B_Sonc_10	1,3,5-Trinitrobenzene	99-35-4	0.100	0.0138	0.0400	mg/Kg	80	116	20	80	116	20		
		1,3-Dinitrobenzene	99-65-0	0.100	0.0166	0.0400	mg/Kg	73	119	20	73	119	20		
		2,4,6-Trinitrotoluene	118-96-7	0.100	0.0307	0.100	mg/Kg	71	120	20	71	120	20		
		2,4-Dinitrotoluene	121-14-2	0.100	0.0147	0.0400	mg/Kg	75	121	20	75	121	20		
		2,6-Dinitrotoluene	606-20-2	0.100	0.0191	0.0400	mg/Kg	79	117	20	79	117	20		
		2-Amino-4,6-dinitrotoluene	35572-78-2	0.100	0.0329	0.100	mg/Kg	71	123	20	71	123	20		
		4-Amino-2,6-dinitrotoluene	19406-51-0	0.100	0.0299	0.100	mg/Kg	64	127	20	64	127	20		
		HMX	2691-41-0	0.100	0.0227	0.0400	mg/Kg	74	124	20	74	124	20		
		m-Nitrotoluene	99-08-1	0.200	0.0640	0.100	mg/Kg	67	129	20	67	129	20		
		Nitrobenzene	98-95-3	0.300	0.0850	0.100	mg/Kg	67	129	20	67	129	20		
		Nitroglycerin	55-63-0	2.00	0.215	0.400	mg/Kg	73	124	20	73	124	20		
		o-Nitrotoluene	88-72-2	0.200	0.0472	0.100	mg/Kg	70	124	20	70	124	20		
		PETN	78-11-5	2.00	0.493	1.00	mg/Kg	72	128	20	72	128	20		
		Picric acid	88-89-1	0.100	0.0563	0.100	mg/Kg	38	154	20	38	154	20		
		p-Nitrotoluene	99-99-0	0.200	0.0365	0.100	mg/Kg	71	124	20	71	124	20		
		RDX	121-82-4	0.200	0.0430	0.100	mg/Kg	67	129	20	67	129	20		
		Tetryl	479-45-8	0.200	0.0439	0.100	mg/Kg	68	135	20	68	135	20		
		1,2-Dinitrobenzene	528-29-0	0.100	0.0190	0.0400	mg/Kg							78	119

8330B_DOD5	8330B_Sonc_10	1,3,5-Trinitrobenzene	99-35-4	0.100	0.0138	0.0400	mg/Kg	80	116	20	80	116	20		
		1,3-Dinitrobenzene	99-65-0	0.100	0.0166	0.0400	mg/Kg	73	119	20	73	119	20		
		2,4,6-Trinitrotoluene	118-96-7	0.100	0.0307	0.100	mg/Kg	71	120	20	71	120	20		
		2,4-Dinitrotoluene	121-14-2	0.100	0.0147	0.0400	mg/Kg	75	121	20	75	121	20		
		2,6-Dinitrotoluene	606-20-2	0.100	0.0191	0.0400	mg/Kg	79	117	20	79	117	20		
		2-Amino-4,6-dinitrotoluene	35572-78-2	0.100	0.0329	0.100	mg/Kg	71	123	20	71	123	20		
		4-Amino-2,6-dinitrotoluene	19406-51-0	0.100	0.0299	0.100	mg/Kg	64	127	20	64	127	20		
		HMX	2691-41-0	0.100	0.0227	0.0400	mg/Kg	74	124	20	74	124	20		
		m-Nitrotoluene	99-08-1	0.200	0.0640	0.100	mg/Kg	67	129	20	67	129	20		
		Nitrobenzene	98-95-3	0.300	0.0850	0.100	mg/Kg	67	129	20	67	129	20		
		Nitroglycerin	55-63-0	2.00	0.215	0.400	mg/Kg	73	124	20	73	124	20		
		o-Nitrotoluene	88-72-2	0.200	0.0472	0.100	mg/Kg	70	124	20	70	124	20		
		PETN	78-11-5	2.00	0.493	1.00	mg/Kg	72	128	20	72	128	20		
		Picric acid	88-89-1	0.100	0.0563	0.100	mg/Kg	38	154	20	38	154	20		
		p-Nitrotoluene	99-99-0	0.200	0.0365	0.100	mg/Kg	71	124	20	71	124	20		
		RDX	121-82-4	0.200	0.0430	0.100	mg/Kg	67	129	20	67	129	20		
		Tetryl	479-45-8	0.200	0.0439	0.100	mg/Kg	68	135	20	68	135	20		
		1,2-Dinitrobenzene	528-29-0	0.100	0.0190	0.0400	mg/Kg							78	119

Moisture		Percent Moisture	STL00177	0.100		0.0500	%								
		Percent Solids	STL00234	0.100		0.0500	%								

TA Denver Limits - Water

Method Code	Prep Method	Analyte Description	CAS Number	RL	MDL	LOD	Units	LCS - Low	LCS - High	LCS - RPD %	MS - Low	MS - High	MS - RPD %	Surrogate Low	Surrogate High
8330A	3535	Tetryl	479-45-8	0.240	0.0793	0.200	ug/L	64	128	30	64	128	30		
		PETN	78-11-5	2.00	0.416	1.20	ug/L	73	127	30	73	127	30		
		Nitroglycerin	55-63-0	3.00	0.921	2.00	ug/L	74	127	30	74	127	30		
		Nitrobenzene	98-95-3	0.400	0.0910	0.200	ug/L	65	134	30	65	134	30		
		RDX	121-82-4	0.200	0.0523	0.120	ug/L	68	130	30	68	130	30		
		p-Nitrotoluene	99-99-0	1.00	0.200	0.400	ug/L	71	127	30	71	127	30		
		m-Nitrotoluene	99-08-1	0.400	0.0834	0.200	ug/L	73	125	30	73	125	30		
		1,3,5-Trinitrobenzene	99-35-4	1.00	0.200	0.400	ug/L	73	125	30	73	125	30		
		1,3-Dinitrobenzene	99-65-0	0.400	0.0887	0.200	ug/L	78	120	30	78	120	30		
		2,4,6-Trinitrophenol	88-89-1	0.400	0.0436	0.120	ug/L	73	124	30	73	124	30		
		2,4,6-Trinitrotoluene	118-96-7	0.400	0.0724	0.200	ug/L	71	123	30	71	123	30		
		2,4-Dinitrotoluene	121-14-2	0.400	0.0838	0.200	ug/L	78	120	30	78	120	30		
		2,6-Dinitrotoluene	606-20-2	0.200	0.0645	0.200	ug/L	77	127	30	77	127	30		
		2-Amino-4,6-dinitrotoluene	35572-78-2	0.200	0.0507	0.120	ug/L	79	120	30	79	120	30		
		4-Amino-2,6-dinitrotoluene	19406-51-0	0.200	0.0577	0.120	ug/L	76	125	30	76	125	30		
		HMX	2691-41-0	0.400	0.0876	0.200	ug/L	65	135	30	65	135	30		
		o-Nitrotoluene	88-72-2	0.400	0.0855	0.200	ug/L	70	127	30	70	127	30		
		1,2-Dinitrobenzene	528-29-0	0.200	0.0500	0.120	ug/L	83	119	30	83	119	30	83	119
6010C	3010A	Aluminum	7429-90-5	300	18.0	70.0	ug/L	86	115	20	86	115	20		
		Antimony	7440-36-0	20.0	5.17	12.0	ug/L	88	113	20	88	113	20		
		Arsenic	7440-38-2	25.0	4.41	15.0	ug/L	87	113	20	87	113	20		
		Barium	7440-39-3	10.0	0.820	2.00	ug/L	88	113	20	88	113	20		
		Beryllium	7440-41-7	1.50	0.474	1.20	ug/L	89	112	20	89	112	20		
		Cadmium	7440-43-9	5.00	0.452	1.80	ug/L	88	113	20	88	113	20		
		Calcium	7440-70-2	1000	34.5	135	ug/L	87	113	20	87	113	20		
		Chromium	7440-47-3	15.0	0.663	2.60	ug/L	90	113	20	90	113	20		
		Cobalt	7440-48-4	15.0	1.23	4.50	ug/L	89	114	20	89	114	20		
		Copper	7440-50-8	15.0	4.20	10.0	ug/L	86	114	20	86	114	20		
		Iron	7439-89-6	100	22.0	85.0	ug/L	87	115	20	87	115	20		
		Lead	7439-92-1	15.0	2.74	10.0	ug/L	86	113	20	86	113	20		
		Magnesium	7439-95-4	500	10.7	40.0	ug/L	85	113	20	85	113	20		
		Manganese	7439-96-5	10.0	0.264	1.00	ug/L	90	114	20	90	114	20		
		Nickel	7440-02-0	40.0	2.56	5.00	ug/L	88	113	20	88	113	20		
		Potassium	7440-09-7	3000	237	940	ug/L	86	114	20	86	114	20		
		Selenium	7782-49-2	22.0	4.86	19.0	ug/L	83	114	20	83	114	20		
		Silver	7440-22-4	15.0	0.933	3.50	ug/L	84	115	20	84	115	20		
		Sodium	7440-23-5	5000	117	350	ug/L	87	115	20	87	115	20		
		Strontium	7440-24-6	10.0	0.300	1.20	ug/L	90	113	20	90	113	20		
		Thallium	7440-28-0	40.0	4.91	19.0	ug/L	85	114	20	85	114	20		
		Vanadium	7440-62-2	15.0	1.11	4.00	ug/L	90	111	20	90	111	20		
		Zinc	7440-66-6	150	4.53	15.0	ug/L	87	115	20	87	115	20		
6020A	3020A	Antimony	7440-36-0	6.00	0.400	1.00	ug/L	85	117	20	85	117	20		
		Arsenic	7440-38-2	5.00	0.330	1.00	ug/L	84	116	20	84	116	20		
		Barium	7440-39-3	3.00	0.290	0.950	ug/L	86	114	20	86	114	20		
		Beryllium	7440-41-7	1.00	0.0800	0.300	ug/L	83	121	20	83	121	20		
		Cadmium	7440-43-9	1.00	0.265	1.00	ug/L	87	115	20	87	115	20		
		Chromium	7440-47-3	10.0	0.500	1.80	ug/L	85	116	20	85	116	20		
		Cobalt	7440-48-4	1.00	0.0540	0.200	ug/L	86	115	20	86	115	20		
		Copper	7440-50-8	2.00	0.560	1.80	ug/L	85	118	20	85	118	20		
		Lead	7439-92-1	3.00	0.180	0.700	ug/L	88	115	20	88	115	20		
		Manganese	7439-96-5	3.50	0.310	0.950	ug/L	87	115	20	87	115	20		
		Molybdenum	7439-98-7	2.00	0.140	0.500	ug/L	83	115	20	83	115	20		
		Nickel	7440-02-0	3.00	0.300	1.00	ug/L	85	117	20	85	117	20		
		Selenium	7782-49-2	5.00	0.700	2.00	ug/L	80	120	20	80	120	20		
		Silver	7440-22-4	5.00	0.0330	0.100	ug/L	85	116	20	85	116	20		
		Thallium	7440-28-0	1.00	0.0500	0.200	ug/L	82	116	20	82	116	20		
		Vanadium	7440-62-2	6.00	0.500	2.00	ug/L	86	115	20	86	115	20		
		Zn	7440-66-6	20.0	2.00	8.00	ug/L	83	119	20	83	119	20		

Texas Commission on Environmental Quality

NELAP-Recognized Laboratory Accreditation is hereby awarded to

SGS Accutest - Orlando

4405 Vineland Road, Suite C-15
Orlando, FL 32811-5803

in accordance with Texas Water Code Chapter 5, Subchapter R, Title 30 Texas Administrative Code Chapter 25, and the National Environmental Laboratory Accreditation Program.

The laboratory's scope of accreditation includes the fields of accreditation that accompany this certificate. Continued accreditation depends upon successful ongoing participation in the program. The Texas Commission on Environmental Quality urges customers to verify the laboratory's current location(s) and accreditation status for particular methods and analyses (www.tceq.texas.gov/goto/lab). Accreditation does not imply that a product, process, system or person is approved by the Texas Commission on Environmental Quality.

Certificate Number: T104704404-17-10
Effective Date: 6/1/2017
Expiration Date: 5/31/2018

Executive Director Texas Commission on Environmental Quality

Texas Commission on Environmental Quality

NELAP - Recognized Laboratory Fields of Accreditation

Certificate: T104704404-17-10

SGS Accutest - Orlando
Expiration Date: 5/31/2018

4405 Vineland Road, Suite C-15
Orlando, FL 32811-5803
Issue Date: 6/1/2017

These fields of accreditation supercede all previous fields. The Texas Commission on Environmental Quality urges customers to verify the laboratory's current accreditation status for particular methods and analyses.

Matrix: ***Non-Potable Water***

Method EPA 1010

Analyte	AB	Analyte ID	Method ID
Ignitability	FL	1780	10116606

Method EPA 120.1

Analyte	AB	Analyte ID	Method ID
Conductivity	FL	1610	10006403

Method EPA 1664

Analyte	AB	Analyte ID	Method ID
n-Hexane Extractable Material (HEM) (O&G)	FL	1803	10127807

Method EPA 180.1

Analyte	AB	Analyte ID	Method ID
Turbidity	FL	2055	10011606

Method EPA 200.7

Analyte	AB	Analyte ID	Method ID
Aluminum	FL	1000	10013806
Antimony	FL	1005	10013806
Arsenic	FL	1010	10013806
Barium	FL	1015	10013806
Beryllium	FL	1020	10013806
Cadmium	FL	1030	10013806
Calcium	FL	1035	10013806
Chromium	FL	1040	10013806
Cobalt	FL	1050	10013806
Copper	FL	1055	10013806
Iron	FL	1070	10013806
Lead	FL	1075	10013806
Magnesium	FL	1085	10013806
Manganese	FL	1090	10013806
Molybdenum	FL	1100	10013806
Nickel	FL	1105	10013806
Potassium	FL	1125	10013806